JULY, 1958
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FT food technology

Aksel G. Olsen
1958 President for 1958-1959
and the
Executive Committee of the Council
in Session at the
18th Annual Meeting in Chicago

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IN THIS ISSUE—

THE TECHNICAL ARTICLES IN BRIEF

Packaging *Pressure propelled foods*

The five articles that open the technical section comprised the session on pressurized foods (18th Annual Meeting, IFT, Chicago, May 27, 1958). The articles cover the history of pressurized foods, the packaging requirements, and factors in planning a pressurized food manufacturing program. (See pages 317-337).

Bacteriological evaluation *Citrus juices*

Enterococcus-like organisms in citrus juices. Roger Patrick and E. C. Hill.

About 71% (244) of the samples of citrus juices analyzed for bacterial contamination contained streptococcus-like microorganisms; 11% (26) of the cultures survived the presumptive test for enterococci and about 5.5% survived the confirmatory test. Seven cultures were believed similar to *S. liquefaciens*, even though they failed to survive the heat test of 62° C. for 30 minutes, failed to coagulate milk in the presence of 0.1% methylene blue but did reduce the indicator, did not produce acid in litmus milk, and did not impart a bitter taste to the proteolyzed casein. The remaining 6 cultures failed to grow at pH 9.6 at 37° C. and also failed to coagulate milk in the presence of 0.1% methylene blue. These two irregularities were observed when comparing the characteristics of these cultures with *S. faecalis*. Four of the 6 cultures were isolated from juice of cold-damaged fruit extracted in the Citrus Experiment Station's pilot plant where sanitation is exceptionally good. Streptococcus-like organisms were eliminated from juice readily by heating for 14 seconds to 165° F. Commercial concentrates could have been reinfected with single strength juice contaminated from the cooling plates of the heat exchanger while in the preparation of a 42° Brix product from juice of a higher concentration. (See page 337).

Biochemical evaluation *Shrimp quality*

Biochemical methods for determining shrimp quality. I. Study of analytical methods. Marcel Gagnon and Carl R. Fellers.

Biochemical, organoleptic and bacteriological methods were studied in relation to developing an objective test for estimating the degree of freshness in frozen breaded shrimp. Moisture and breading content along with total bacteria count, taste panel measurement, total nitrogen and volatile bases were determined on each sample. A quality scale based on the ratio values obtained was proposed to estimate objectively the quality of frozen breaded shrimp. The proposed quality scale was based on chemical tests which could be quicky carried out with a minimum of labor and simple equipment available in any small laboratory. No special technical training is necessary. For routine testing of relatively homogeneous samples of shrimp, only the volatile bases content need be determined. Total protein content varies in a narrow range. Thus, for plant quality control, volatile bases furnish valuable qualitative results. (See page 340).

Biochemical evaluation *Shrimp quality*

Biochemical methods for determining shrimp quality. II. Survey of market frozen breaded shrimp. Marcel Gagnon and Carl R. Fellers.

Frozen breaded shrimp quality was surveyed at the retail store level by means of bacteriological and chemical tests. A total of 144 samples was used. Results showed that the ratio test (VB/TN) was reliable and realistic even in heterogeneous samples. Total nitrogen was found to have the greatest influence on the variations of the volatile base. Moisture and breading content, though playing a part in those variations, could not be found significantly responsible. Analysis of the distribution revealed that the distribution of the ratio values was almost symmetrical, indicating that the ratio values were reliable. The ratio test can be used as a quality index within the limit of the proposed quality zones for frozen breaded shrimp. (See page 344).

Stability evaluation *Tomato paste*

Objective criteria for storage changes in tomato paste. B. S. Luh, Sherman Leonard, and G. L. Marsh.

Tomato paste made by the hot-break process and containing 26% total solids was stored at temperatures varying from 6° C. (42.8° F.) to 55° C. (131° F.) for 15 months. Samples were tested for changes in chemical constituents. Results indicate that tomato paste deteriorates rapidly at temperatures higher than 25° C. (77° F.). Serum color was found to darken, and increases in hydroxymethyl furfural and tannin-like substances occurred. High storage temperature and a long storage period also contributed to decreases in amino nitrogen, ascorbic acid, and vacuum. Among these, determination of serum color and hydroxymethyl furfural content were found to be promising as objective criteria for evaluating quality of tomato paste. To maintain quality, storage at low temperature, preferably below 25° C. (77° F.), is desirable. The lower the storage temperature, the slower the rate of chemical deterioration. Objective tests might be useful as supplements to the prevailing methods of grading tomato paste. Effect of storage temperature on shelf-life and chemical composition of tomato paste is discussed. (See page 347).

JULY COVER. The cover cut for July presents Dr. Aksel G. Olsen, IFT president for 1958-1959, and the Executive Committee of the Council, both members and invited participants, in action at their meeting of May 24, 1958, Palmer House, Chicago, Dr. Emil Mrak, IFT president, 1957-58, presiding. Clockwise around the table beginning, left, with Dr. Robert M. Schaffner, who reported the Monticello, Illinois, educational conference, are, next in line, Arthur N. Prater, Marian A. Gilchrist, Ida Gerber, John C. Ayres, Philip K. Bates (IFT president, 1954-55), Colonel C. S. Lawrence, Ray B. Wakefield (President-Elect), Emil M. Mrak, Aksel G. Olsen, John H. Nair, George F. Garnatz (IFT president, 1956-57), Loren B. Sjöström, Martin S. Peterson, and W. F. Robertson (visitor).

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FT food technology

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Quality evaluation *Deep-fat fried peas*

Quality evaluation of deep-fat fried peas. W. Sathiraswasti and D. K. Salunkhe.

Various objective tests were conducted to determine the proper maturity of peas for deep-fat frying. The process of deep-fat frying was developed. Chemical analyses were conducted to determine the nutritive value of the product. Organoleptic appraisal was also made. Peas were fried in cocoanut oil (Dri-Fri) at 350° F. Proper frying technique was developed for the experiments. The protein content of deep-fat fried peas increased whereas fat and moisture contents decreased as the size of the peas increased. The over-mature peas (size 6) when deep-fat fried had better "eye appealing" color than that of immature deep-fat fried peas. The taste of the deep-fat fried peas was directly correlated with the size of raw peas. The findings of this study may be helpful to the canning industry since over-mature peas are of little value for processing purposes. (See page 351).

Consistency *Tomato paste*

Observations on the consistency of tomato paste. C. J. B. Smit and B. K. Nortje.

San Marzano and Pearson tomatoes were manufactured into paste by using two different screen sizes. Some fruit samples were immediately heated to 180° F. after breaking; others were delayed for three hours at 70–80° F. between breaking and preheating. A larger screen size in the finisher gave a paste with a higher consistency. Delay between breaking and preheating gave a higher consistency at the same total soluble solids level than samples which were immediately preheated after breaking, even though the calcium pectate, methoxyl content and comparative serum viscosities were lower. As the water insoluble solids content was rather similar for the two treatments, it is suggested that the difference in consistency was due to a qualitative difference in the insoluble substances. (See page 356).

Yeast technology *Activated dry yeast*

Factors affecting loss of nitrogen and fermenting power of rehydrated active dry yeast. R. K. Sant and W. H. Peterson.

Active dry yeast (ADY) lost about twice as much nitrogen when reconstituted at 4.5° C. as at 37° C. The excessive loss at 4.5° C. could not be prevented by reconstituting cells in concentrated extracts of the cells or in minimal quantities of water containing coenzymes, pectin, calcium chloride or high levels of glucose. Vapor rehumidification of ADY decreased loss of nitrogen on subsequent reconstitution. A moisture content of about 25% appears to be the critical level for protection against cell damage by cold water. Suspension of ADY in water at 4.5° C. killed about 95% of the cells as judged by plate counts and reduced their CO_2-producing ability about 90% below that of samples rehydrated at 37° C. If the moisture content of ADY is raised to 25% by vapor rehumidification, suspension in water at 4.5° C. has no ill effects on number of viable cells or their CO_2-production. Tests at 26° C. gave results more nearly like those at 37° C. Full activity was reached at a moisture content of about 17%. (See page 359).

New product *Pineapple juice powder*

Pineapple juice powder. G. K. Notter, D. H. Taylor, and J. E. Brekke.

Pineapple-juice powder of good quality is produced from commercial concentrate by drying at 150° F. at 2 mm. Hg pressure for 2 hours and 45 minutes in a vacuum shelf drier. Product is packaged with an in-package desiccant and stored at progressively higher temperatures up to 90° F. as the moisture content of the powder decreases. The addition of SO_2 to the concentrate protected the flavor of powders stored at 90° F. If the product is stored at temperatures of 70° to 77° F., there is no need for addition of SO_2 or other preservative, since the flavor and color changes after one year are negligible. Under conditions of the experiments reported, vacuum packaging was not found necessary for retention of flavor. (See page 363).

Freezing technology *Poultry meats*

The effect of various freezing methods on quality of poultry meat. W. W. Marion and W. J. Stadelman.

Chicken fryers, fowl, turkey fryers and mature turkey toms were frozen by four different methods. Subsequent quality of meat was evaluated by physical measurements of percentage drip, percentage total cooking loss, and tenderness of the *pectoralis major* muscle. Freezing methods used were: (1) liquid freezer adjusted to 10° F. (-11° C.) and 50 feet per minute flow; (2) liquid freezer for 15 minutes, after which the product was transferred to a still-air plate-type freezer at -5° F. (-19° C.) ; (3) Still-air plate-type freezer alone and, (4) a commercial freezer, -15° F. (-26° C.) with air circulation of 150 feet per minute. Method of freezing was found to exert no significant effect on any of the measured variables. In the case of chicken fryers, there was a significant difference in tenderness between left and right *pectoralis major* muscles. This difference could not be adequately explained. A larger number of individuals selected one of the two darkest colored turkeys as first choice in the color preference study. There was greater rejection of the lighter colored turkeys except for the whitest one which was one of the most frequently selected turkeys. Its very white color connoted to some individuals additional "cleanliness" or "sanitation." (See page 367).

Bacteriological evaluation

Pre-cooked frozen foods

Bacteriological evaluation of marketed precooked frozen foods in relation to public health. A. Doreen Ross and F. S. Thatcher.

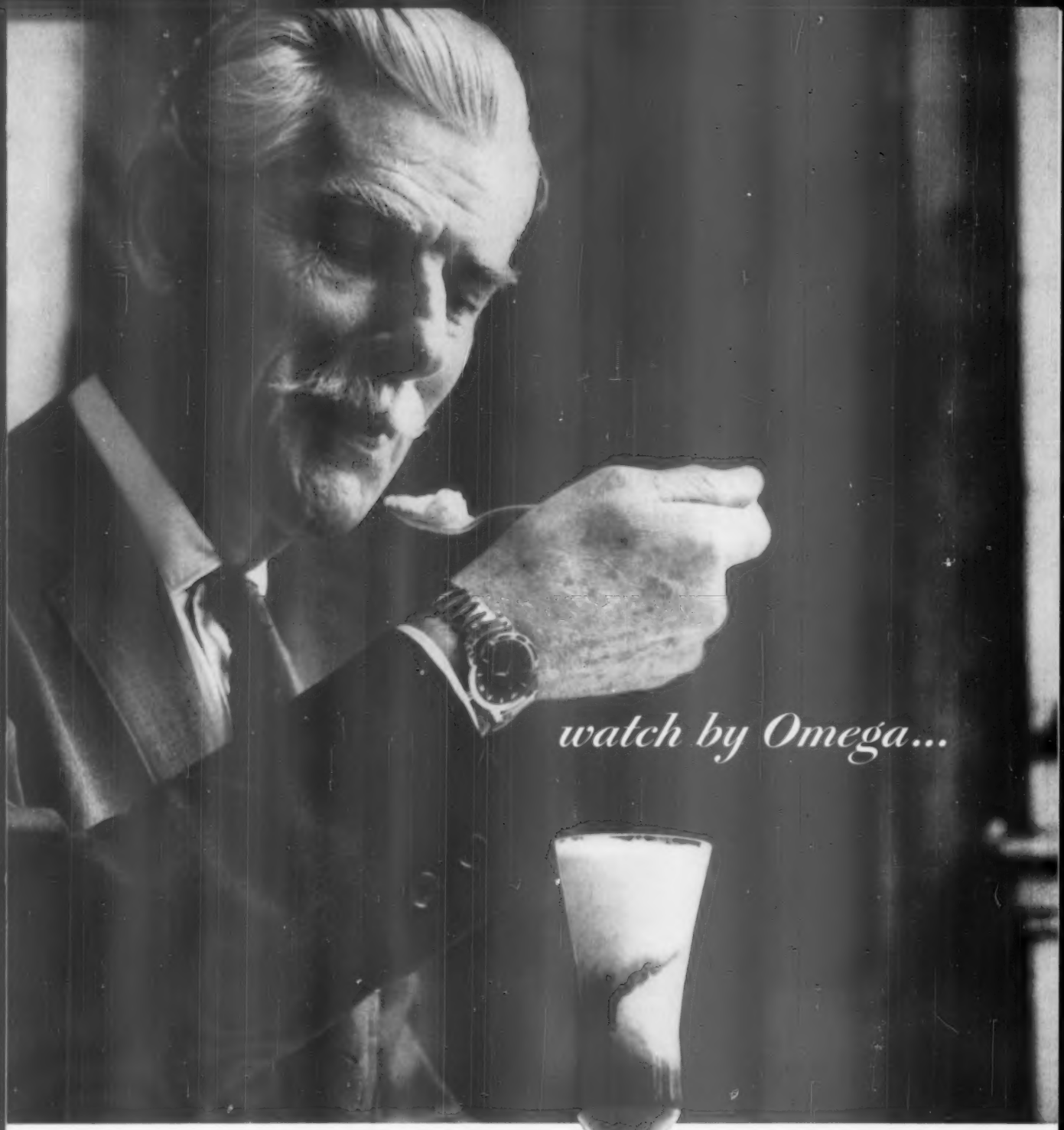
Bacteriological examination of 117 specimens of precooked frozen foods purchased from retail outlets has established that a proportion of these foods are highly contaminated. Certain specimens were shown to contain *E. coli*, coagulase-positive staphylococci and enterococci in large numbers. Members of the Bethesda-Ballerup and Providence groups of the paracolon organisms were found. The requirement of exacting standards of hygiene in the source factories was deduced. (See page 369).

Flavor improvement *Monosodium glutamate*

The influence of added monosodium glutamate on the flavor of processed green beans. Lois A. Sather, L. A. Pettit, and R. W. Hirzel.

After approximately 3, 6, and 14 months' storage, color and flavor evaluation tests were made on canned and frozen bush and pole green beans and bush wax beans containing no added MSG, a low level of added MSG (approximately 0.125% by weight), and a higher level of added MSG (approximately 0.200% by weight). All samples were prepared by commercial procedures. In canned samples, MSG was added after salt and beans had been measured into the cans. In the frozen samples, MSG was added just prior to sealing the package. The beans were not preselected for quality but were regular plant run.

Readings from the Hunter Color Difference Meter indicated no difference in color between MSG treated and untreated canned and frozen green bush and pole beans and



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bush wax beans after 3, 6, and 14 months' storage. Under conditions of this test, large panels (94 to 142 tasters) of untrained student tasters indicated no consistent flavor preferences for the MSG treated or untreated bean samples. The student tasters could detect a difference in flavor between the MSG treated and the untreated beans but could not as a group consisently agree on preference. (See page 372).

Horticultural factors *Canning quality*

Influence of ripening temperature, ripeness level, and growing area on quality of canned Bartlett pears. L. L. Claypool, Sherman Leonard, B. S. Luh, and Marion Simone.

The influence of ripening temperature, ripeness level, and growing area on chemical composition and flavor of California canned Bartlett pears was investigated. Fruits from different growing areas differ in acidity, soluble solids, and volatile reducing substances. Pears ripened to a firmness of about 2 pounds prior to canning were of better flavor than those ripened and canned at greater firmness. Pears ripened at a temperature of 68° F. (20° C.) resulted in a canned product which was superior in flavor to those ripened at 77° F. (25° C.) and 86° F. (30° C.), provided they were canned at the same average pressure test of two pounds. Firmness at canning and ripening temperature are two important factors greatly influencing flavor of the canned product. Growing area has a lesser influence on the quality of the canned product. (See page 379).

FUTURE MEETINGS FOR FOOD TECHNOLOGISTS

June 9–August 29	Gordon Research Conferences, Colby Junior College, New London, New Hampshire; New Hampton School, New Hampton; New Hampshire and Kimball Union Academy, Meriden, New Hampshire.
August 5–8	Poultry Science Association Meeting, Cornell University, Ithaca, New York
September 21–28	Eleventh World's Poultry Congress, Mexico City, Mexico
October 13–15	Association of Official Agricultural Chemists, Inc., Shoreham Hotel, Washington, D. C.
October 22–24	Fifth National Vacuum Symposium, American Vacuum Society, Sir Francis Drake Hotel, San Francisco, California
December 26–31	American Association for the Advancement of Science, Washington, D. C.

1959	
May 17–21	**Nineteenth Annual Meeting of the Institute of Food Technologists, Bellevue-Stratford Hotel, Philadelphia, Pennsylvania**
1960	
May 15–19	**Twentieth Annual Meeting of the Institute of Food Technologists, Civic Auditorium and Hotel Whitcomb, San Francisco, California**
1961	
May 7–11	**Twenty-first Annual Meeting of the Institute of Food Technologists, Hotel Statler, New York, New York**
1962	
June 17–20	**Twenty-second Annual Meeting of the Institute of Food Technologists, Americana Hotel, Bal Harbor, Miami Beach, Florida**

An open invitation is extended to readers of FOOD TECHNOLOGY Illinois notices of annual or national meetings of interest to food to send in to the Editorial Office, 11606 South Bell Avenue, Chicago 43, technologists.

Enzymatic Oxygen Removal from Packaged Foods*

Don Scott
Fermco Laboratories Inc., Chicago

NEXT TO microbiological deterioration, the most widespread deteriorative factor confronting the modern food technologist is the presence of oxygen.

Not only is oxygen responsible for the oxidative rancidity occurring in fat-containing foods but it is also responsible for such diverse changes as the loss of color of strawberry jam, the discoloration of luncheon meats under fluorescent lights, the development of the varnish-like terpene flavor in orange soda, and even the dark grey-brown discoloration that occurs in canned dog food. In non-foods, oxygen plus moisture may cause corrosion or rust formation as in the metals.

Modern food processing has only served to accentuate the problems caused by oxygen. Frozen peaches brown enzymatically through the action of polyphenoloxidase during thawing—a problem not encountered with canned peaches. Dried foods have unique problems due to the concentration of the reactants in drying so that Maillard browning, insolubilization, loss of original color, and development of off odors and flavors result. Even such foods as dried carrots offer a particularly sensitive storage problem. Cheese sold in presliced form—particularly natural cheeses—tend to mold in the presence of oxygen; prepeeled potatoes discolor. The developing trend to irradiated foods is likewise bringing with it new oxygen problems. There is a definite correlation between the off flavors developed during irradiation of milk and the presence of oxygen.

All this tends to place oxygen—and antioxidants—in a new prominence. In this instance, fortunately, the technology has followed closely behind the problems and the means now appears to be at hand to solve many of these oxygen problems.

Aside from physical means of reducing oxygen content, many chemical antioxidants—both synthetic and naturally formed—are used.

Antioxidants may operate in various ways, and may be classified according to the mode of action into the following categories:

TYPES OF ANTIOXIDANTS

Type	*Examples*	*Application*
Phenolics (chainbreakers)	BHA, BHT, NDGA	Fats and oils
Sequestrants	Citric acid	Fats and oils
	Ascorbic acid	Fats and oils
Reducing agents	Ascorbic acid	Aqueous products
Specific O_2 scavengers	Fermcozyme	Aqueous products
	OxyBan	Fats, oils, aq. and dry prod.

Phenolics. Phenolics are used in fats and oils and fat-containing foods to slow down oxidative rancidity. It is generally believed that in the oxidative deterioration of fats, a fat molecule reacts with oxygen to form an activated peroxide, and those activated peroxide molecules tend to act as catalysts for the oxidation by making a chain reaction possible. These hydroperoxides break down to form unpalatable and foul smelling aldehydes, ketones and acids. It is, of course, well known that oxidative rancidity is an autocatalytic reaction. Phenolic antioxidants function by combining with the hydroperoxides formed in the early stages of oxidative rancidity thus breaking the chain reaction and slowing the rate of oxidation. The most commonly used phenolics are BHT, BHA, NDGA, and propyl gallate. It is important to realize that phenolics slow oxidative rancidity, but do not prevent it; they cannot even begin to function until rancidity has taken place to a slight extent. Phenolics will not halt oxidative rancidity if it has progressed to a substantial peroxide value as the peroxides already formed are by then in stoichiometric excess of the small amount of phenolic added and sufficient excess peroxide remains to speed the oxidation. Phenolics are without effect on hydrolytic rancidity catalyzed by the enzyme lipase, nor are they effective against enzymatic fat oxidation catalyzed by the enzyme lipoxidase.

Sequestrants. Sequestrants function by binding the metal ions such as copper that tend to catalyze oxidative rancidity. Agents such as citric acid are usually combined with the chain-breaking phenolic antioxidant to give a synergistic effect. By themselves sequestering agents are relatively ineffective as antioxidants except where the product has been processed within copper equipment and a high metal ion level exists.

General Reducing Agents. Used because of the obvious fact that reduction or elimination of oxygen within the package will minimize or eliminate oxidative problems, general reducing agents function by being readily oxidizable. Hence, in theory they are oxidized by the oxygen present before the food itself is affected and tend to protect the food. These agents are generally applicable only to aqueous systems or emulsions.

The most widely used general reducing agent is ascorbic acid. In certain instances, the use of ascorbic acid has definite disadvantages. To be effective in beer, for example, so much must be used that it affects the flavor. I have been told that even one gram in a

* Address delivered by Dr. Don Scott, Fermco Laboratories, Inc., 4941 South Racine Avenue, Chicago 9, Illinois, at the Ohio Valley Section of the IFT Meeting on January 10, 1958 in Columbus, Ohio and also at the Quartermaster Food and Container Institute, March 17, 1958, Chicago, Ill. Dr. Scott is an Associate Editor of *Food Technology*.

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barrel of beer can be detected by its flavor effect. The excess ascorbic acid, over and above what is needed to react with the oxygen can and often does react with other ingredients in the food and for example, causes "skunkiness" in beer. Similarly, certain azo food colors such as amarinth, sunset yellow, tartrazine, and orange No. 1 are bleached by ascorbic acid. Thus, while ascorbic acid is effective in preventing the corrosion of the can by canned grape soda the excess ascorbic acid has been known to turn grape soda a bright blue or green, depending on the dyes used.

Enzymatic Oxygen Scavengers. The newest type of antioxidant—an antioxidant essentially specific for oxygen—is the enzymatic O_2 scavenger. The ability of glucose oxidase to take up oxygen was first noted in 1904 in ground mold mycelium. As used today, the enzyme is applied as a system consisting of glucose oxidase and catalase. This enzyme system is derived from a mold grown under conditions very similar to the procedures used for the production of antibiotics. The mold mycelium containing the enzyme system is filtered off, the mold mycelium disrupted, and the enzyme in solution separated from the cell walls by filtration or centrifugation.

The enzyme solution is then put through several purification steps to remove impurities. Then, the enzyme is precipitated from the aqueous solution by the addition of a water miscible solvent such as acetone or ethanol. The precipitated enzyme is filtered or centrifuged off, dried, then extracted with buffer, standardized, and stabilizers added. The final product is a clear amber solution, stable under refrigeration for several years, and at room temperatures for months. This product, Fermcozyme, is the basic material in enzymatic deoxygenation.

As stated before, Fermcozyme contains both glucose oxidase and catalase, that catalyze, respectively, the following reactions:

(1) $2\ \text{glucose} + 2\ O_2 + 2\ H_2O \longrightarrow 2\ \text{gluconic acid} + 2\ H_2O_2$

(2) $2\ H_2O_2 \longrightarrow 2\ H_2O + O_2$

Thus, the net reaction is:

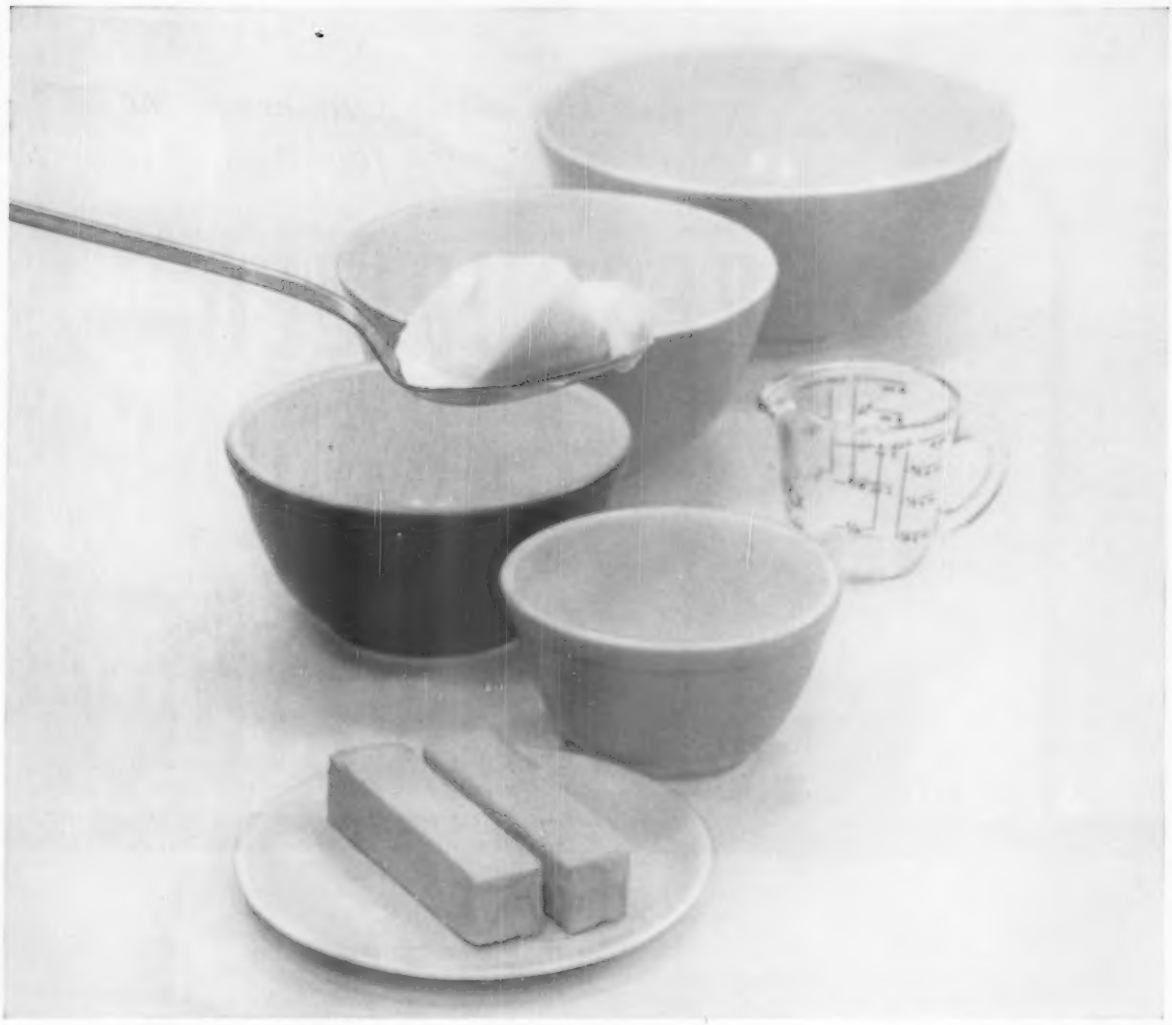
(3) $2\ \text{glucose} + O_2 \longrightarrow 2\ \text{gluconic acid}$

In contrast with the usual chemical antioxidants such as the phenolics, Fermcozyme acts catalytically rather than stoichiometrically and therefore is effective in trace quantities. Furthermore, so long as glucose is present, it will function until all the oxygen has been removed.

Because of the almost absolute specificity of Fermcozyme and other antioxidant preparations based on glucose oxidase-catalase for oxygen and glucose the use of these products is essentially free of side reactions. We say *almost* absolute specificity because, in the presence of ethanol, reaction (2) above does not take place. Instead, the catalase acts as a peroxidase taking the H_2O_2 formed by the oxidase in equation (1) to yield acetaldehyde as follows:

(2a) $H_2O_2 + CH_3CH_2OH \longrightarrow 2\ H_2O + CH_3CHO$

Thus, in the presence of ethanol, the net reaction becomes,



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(3a) glucose + ethanol + O_2 ⟶ gluconic acid + acetaldehyde + H_2O

It is believed that the above reaction takes place in beer and results in the slightly sweet temporary change in flavor of glucose oxidase-catalase treated beer.

ATTRIBUTES OF IDEAL ANTIOXIDANT

The ideal antioxidant may be characterized as follows:

1. Must be non-toxic and non-irritant.
2. Should not impart any foreign odor, or color, or flavor to food.
3. Should not affect nutritive value.
4. Should not conceal inferiority or faulty processing.
5. Should be effective in small quantities and be economical in use.
6. Should be readily applicable to food or to the package.
7. Should preferably be a substance normally in food or naturally formed.
8. Should be specific in its action.

In our opinion, glucose oxidase-catalase is the most ideal antioxidant for increasing the shelf life of sealed packages. No one antioxidant, of course, is the panacea for every off-flavor and discoloration problem caused by oxidation in food products, but because the basis of the antioxidant action of glucose oxidase-catalase is the removal of oxygen the use of this type of antioxidant, should in theory, halt oxidative rancidity even if it has already started or prevent it if it has not taken place. It should also be effective against the other types of degradations in which oxygen is required such as regression of oils, lipoxidase degradation, and even the Maillard reaction.

Glucose oxidase-catalase antioxidants must be used in gas-tight packages. Consequently for some applications, such as in breakfast cereals, the phenolics would seem to be the preferred antioxidant.

APPLICATIONS

Versatility in application is the keynote of the use of Fermcozyme and related products. They can be applied in an extensive variety of ways depending on the nature of the problem. These methods of application (with examples) may be summarized as follows:

A. Aqueous foods
   1. Direct incorporation of Fermcozyme in mayonnaise, soft drinks, flavor concentrates.
   2. Surface treatment as in canned dog food.
   3. Coated on paper or other films for cheese, luncheon meats.
   4. Use of packets of *OxyBan* where it is desirable to keep the enzyme separate from the food.

B. Non-aqueous foods
   1. Direct incorporation of *OxyBan* as in animal feeds.
   2. Use of *OxyBan* packets as in chow mein noodles, and the like.

Let's look at some examples of each of these:

Mayonnaise. Mayonnaise is, by definition, the emulsified semi-solid food prepared from edible vegetable oil, acidifying ingredients, egg yolk-containing ingredients, and spices or flavoring ingredients. In no other fatty food product is oil subjected to so many unfavorable conditions which tend to turn it rancid or to cause it to deteriorate in other ways. As an oil-in-water emulsion the surface area of the oil is very great. In addition the whipping process incorporates approximately 10–12% air into the finished product. Then it is packed in glass containers and exposed to light and stored at ambient temperatures which may be over 100° F. at times.

The mayonnaise industry has made great strides in producing a relatively stable product through such measures as rigid purchase specifications for oil. These specifications prevent the use of oil that has taken up oxygen and would turn rancid rapidly.

The substitution of stainless steel for copper-bearing alloys has also been of great assistance. These earlier precautions of quality of ingredients, particularly the oil, make it possible to greatly extend the shelf-life of mayonnaise by the complete removal of oxygen in the mayonnaise through the use of Fermcozyme.

Early results with Fermcozyme were erratic because of the poor seal obtained with standard closures. These often permitted oxygen to leak into the jar throughout the storage period putting an impossible load on the enzyme. When this closure problem was solved, the experimental work was repeated as follows:

A commercial size batch (60 gals.) of an 80% oil mayonnaise was prepared using frozen egg yolk containing 10% salt as the yolk material and distilled white vinegar as the acidulent. All ingredients came from lots normally used for the commercial production of mayonnaise. An identical batch was made to which 50 p.p.m. Fermcozyme was added. At intervals duplicate samples of each were opened and examined visually and organoleptically by an experienced four-member panel on mayonnaise, and chemically for peroxide value which is generally accepted as a measure of degree of incipient rancidity. Results are given in Table 1.

The removal of occluded oxygen by the glucose oxidase-catalase prevented the development of oxidative

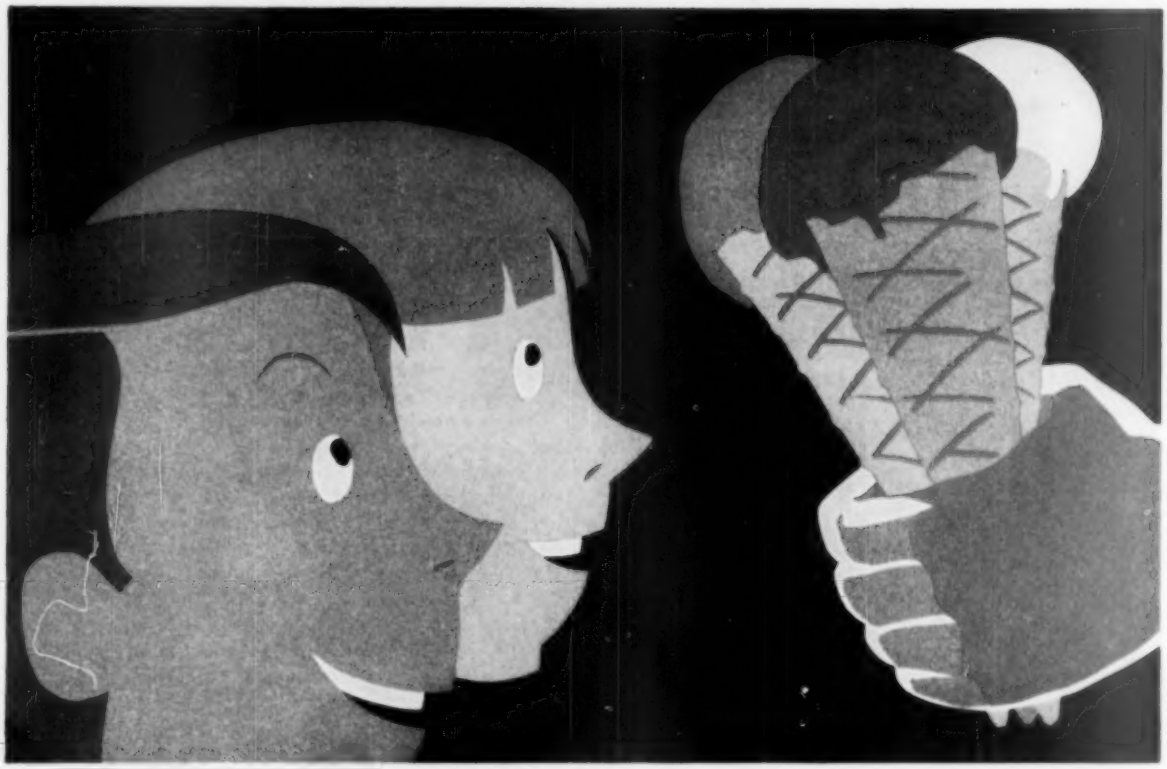
TABLE 1[1,2]

Age months	Organoleptic		Color		Peroxide value mm./kg. (Averages)	
	Control	Fermcozyme treated	Control	Fermcozyme treated	Control	Fermcozyme treated
0	+	+	+	+	0.2	0.2
2	+	+	+	+		
3	—	+	+	+	6.2	0.2
4	—	+	—	+		
5	—	+	—	+		
6	—	+	—	+	14.6	0.8
Legend	+ Fresh — Slightly rancid — Rancid		— Pale yellow + Deep yellow — Very pale yellow			

[1] No physical changes were noted in the experimental samples during the course of the experiment. Shipping movement simulated by carrying samples in the trunk of a car for an extended period of time caused no apparent emulsion breakdown.

[2] From Bloom, J., Scofield, G., and Scott, Don. Oxygen removal prevents rancidity in mayonnaise. *Food Packer*, December, 1956.

(Continued on page 13, after technical section)



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The History and Background of Pressurized Foods*

(Manuscript received May 11, 1958)

W. Earl Graham
Clayton Corporation, St. Louis, Mo.

USE OF THE TERM "aerosol foods" will be assiduously avoided in this discussion. However, in the non-food industries "aerosol" has become a generic term for all pressurized products regardless of whether they are dispensed in the form of a true aerosol, a wet spray, or a foam. In considering the history of pressurized foods, carbonated beverages will be excluded. With carbonated beverages the charging gas is used for its organoleptic properties rather than as a propellent.

Pressurized foods may be defined as products packaged with a non-toxic compressed gas which serves to self-propel the food through a suitably valved closure. In acting as a propellent the gas may alter the physical nature of the food, as it does in whipped cream, or it may, as in chocolate syrup, merely dispense a non-aerated product similar to that packaged by other methods. Whether or not the food is altered on dispensing, the essential function of pressurizing is to make the food available to the consumer in a more convenient form. The aerosol industry learned long ago that if the consumer merely gets the nefarious thrill of "squirting," the product's commercial life will be short lived. Pressurized foods are no exception. In some cases, in addition to dispensing in a more desirable physical form, there must be a time saving, or perhaps better, an "irritation" saving value. Another desirable feature is external cleanliness of the package after the initial use and intermittent storage. For many products, the continuing hermetic seal of the pressurized package may enhance its food conservation value.

ANTECEDENTS AND PRECEDENTS THAT FOUNDED AN INDUSTRY

Pressurized food packages of today have their antecedent in the syphon bottle with which patents covering the use of carbon dioxide date back to 1869 *(11)*. Nitrous oxide was used in a similar container shortly thereafter. The use of nitrogen for pressurized foods was described as early as 1899 *(12)*. A more detailed reference to the usage of these gases has been given previously *(3)*.

During the period 1934–36 whipped cream similar to the present commercial product was produced by packing cream in pint-size metal containers with a suitably sealed-in valve. Nitrous oxide from small auxiliary cartridges was the propellent. Such containers are still used as refillable units for whipped cream by drug store fountains and restaurants.

With the advent of the disposable aerosol container following World War II, the pressurized food industry was on its way. This container resulted from the can manufacturers' adapting aerosol valves to beer and beverage cans. The first pressurized food was whipped cream, and in the early years of production it outnumbered true aerosols, mostly insecticides, more than two to one. It was only when the industry spread to other than true aerosols that the number and volume of pressurized products expanded greatly. In the past 9 years pressurized foods have little more than doubled in production whereas non-foods usage has increased 10- to 15-fold. For 1957 non-food aerosol production was about 400 million units. Pressurized food output—whipped cream and similar toppings—is estimated at about 70 million units. Hundreds of different non-food aerosols are packaged, but the bulk of the volume is in a half dozen products; namely, hair sprays, insecticides, odorant and cologne sprays, shave cremes and paints.

SOME PERSISTENT PROBLEMS

Considering the large potential food market, the lack of development of pressurized foods has been surprising, perhaps more to the aerosol than to the food industry. In a previous report *(4)* approximately 20 food products or types were listed as potential for development. At the same time problems associated with the successful production and marketing were listed. These same problems still prevail and are as follows:

1. Physical modification of foods to permit pressure packaging.
2. Biological stability or preservation factors.
3. Economics of the package.
4. Food laws and regulations.

This symposium will point the way to the solution of many of the above problems. See also *(10)*.

Whipped cream has been named as the foremost pressurized food. There has been a steady growth of non-dairy toppings of a similar nature but with the cream replaced with a vegetable fat. Other food products which have been marketed in a small way are flavoring syrups for home beverages and pancake batter. Within the past year chocolate syrup, barbecue sauce, and cheese flavored dressings for salads have been introduced.

CONTAINERS, VALVES, AND PROPELLENTS

Containers of tinplate required only slight modification of existing beverage containers. Aerosol cans of tinplate and steel are available from the major producers in 4 to 16 fluid ounce capacity. Adequate sealing and interior linings have been developed. Also available are glass (including plastic coated glass),

* Presented at the 18th Annual Meeting of IFT, Chicago, May 27, 1958.

aluminum and plastic (nylon, phenolic, and melamine) containers, mostly, however, in the smaller size ranges *(5)*. In general, since the gas propellents are inert, container corrosion and stability problems are not basically different from those existing for non-pressurized foods. However, since a different container, in part at least, may be used than those in which the food is usually packaged, and the food itself reformulated for pressurizing, the new product system needs to be fully evaluated for container and product stability.

Many of the valves used for non-food aerosols may be utilized with little or no modifications for foods. Basically, valves are classified as to the physical form of the product dispensed, i.e., foam, spray, or liquid. Some metering valves are available *(1)*.

Choice of valve is fully as important as the selection of product formulation and propellent in determining the physical nature of the dispensed product. Thus, by merely changing the "nozzle head" on a pressurized package either a foam or a spray may be dispensed. Valves may be attached to metal containers by a "clinched on" one inch cup or closure, or staked directly to the double-seamed domed top. Suitable means of sealing valves in plastic and glass containers are available.

Early pressurized foods utilized nitrous oxide alone or with a small percentage of carbon dioxide. Within the past year the industry has been stimulated by the art of gassing with nitrogen or other inert gases *(8, 9)*. With such insoluble gases foods can be dispensed in non-aerated form with the same physical characteristics as those poured from other containers. Most of the aerosol gases for foods have been used in packaging before and are fully acceptable from the public health viewpoint *(8)*. Liquefied gas propellents such as fluorinated hydrocarbons are being studied for foods, and it is hoped that full toxicity approval will be obtained. Work has been done on plastic film barriers within the container to permit liquefied gases without intermingling with the food, but has not as yet reached commercial application *(7)*.

SANCTIONED FOR SHIPMENT

Pressurized foods, as are carbonated beverages, are acceptable for shipment under Interstate Commerce Commission and other government regulations. I.C.C. Tariff 10 *(6)*, Section 73.302 provides an exemption under Compressed Gases as follows:

> (a) Compressed gases—when in accordance with—Subparagraph (5) are exempt from specification, marking and labeling requirements. Subparagraph (5) reads: "Foodstuffs or soaps in metal cans with soluble or emulsified compressed gas providing the pressure in the container does not exceed 115 pounds p.s.i. absolute (100 pounds gauge) or 150 pounds p.s.i. absolute at 130° F. The metal containers must be capable of withstanding without bursting a pressure of two times the pressure of contents at 70° F. or one and one-half times the pressure of the contents at 130° F., whichever is greater."
>
> Subparagraph (7) similarly provides for non-soluble compressed gases for metal containers not exceeding 17.6 fl. oz. capacity. At present, containers of all types (i.e., glass, plastic) are entirely exempt from requirements if under 4 fl. oz. capacity or the product is under 40 pounds pressure absolute.

A comparison of internal pressures encountered with packaged beer, carbonated beverages, and pressurized foods is of interest. A carbonated water with 5 volumes of carbon dioxide exerts a gauge pressure of about 72 p.s.i.g. at 70° F. Pressure in bottled or canned beer is normally about 40 p.s.i.g. at 70° F., but during pasteurization may reach 100 p.s.i.g. Pressurized foods are normally charged to about 90 to 100 p.s.i.g. at 70° F.

A discussion of aerosols would not be complete without reference to the scientific work done by the Chemical Specialties Manufacturers Association, whose Aerosol Division has numerous committees correlating cooperative industry efforts on aerosols, including foods. Its *Aerosol Guide (2)* is indispensable to all who are developing or planning to market pressurized products.

CONCLUSION

In conclusion, it can be asserted that the necessary ingredients for the development of pressurized foods are available. These include:

1. An American public anxious to have foods packaged in a convenient, time- or irritation-saving form and a food industry capable of fulfilling this desire.
2. Food technologists with the basic knowledge of foods and the creative imagination to modify them where necessary for pressure packaging.
3. An aerosol industry with the background of pressure containers, valves, propellents, machinery and filling techniques required to complete the package.

The purpose of this symposium is to provide the impetus that these essential ingredients may be blended into successful new products.

ADDENDUM

Glossary of Terms Used with Pressurized Foods

[Adapted from CSMA, Aerosol Guide (2)]

Actuator. A "spray" head or external actuating part of valve mechanism which contains the dispensing orifice or nozzle.

Aerosol. A suspension of fine solid or liquid particles in air or gas, as smoke, fog, or mist. As defined by the Department of Agriculture, 100% of the particles in an insecticidal aerosol spray must have a diameter less than 50 microns and 80% of the particles must have a diameter less than 30 microns.

Aerosol Product. Self-contained sprayable product in which the propellent force is supplied by a liquefied gas. Includes space, residual, surface coating, foam and various other types of products. The term *aerosol* as used here is not confined to the scientific definition.

Container. Metal, glass or plastic shell in which a pressurized formulation is packaged. With suitable adaptation for a valved closure, the container must meet Interstate Commerce Commission requirements for shipment of compressed gases.

Compressed Gas. Any non-toxic, flavor free gas used under pressure in packaging foods. May be soluble or relatively insoluble. If liquid under normal packaging pressures and temperatures, constant pressure will be maintained throughout dispensing life of container. With non-liquefied gases, pressures in the container will drop as the headspace increases.

FLUID FLOW. The product is dispensed in a non-aerated liquid or stream just as if it were poured from a non-pressurized package. To accomplish, the gas must be insoluble in the food or otherwise prevented from intermixing.

FOAM PRODUCT. Pressurized formulation containing a solution or emulsion which is dispensed in a highly fluffy form by a compressed or liquefied gas propellent.

GASSING. Charging of the propellent in the food container during the filling operation. Compressed gases for foods have been "pressure loaded," that is, injected under pressure back through the sealed valve after the food has been filled and container closed. Many non-foods using large percentages of liquefied gases are "cold" or refrigerated filled in which the gas is chilled below its boiling point and liquid filled into the open container before closing.

OVERCAP. Exterior protective cap over valve and actuator to prevent accidental discharge.

PRESSURE. Internal force per unit area exerted by any material. Since the pressure is directly dependent on the temperature, the latter must be specified. The pressure may be reported in either of two ways:

(A) *Absolute pressure*. The total pressure with zero as a reference point. Usually expressed as pounds per square inch absolute (p.s.i.a.).

(B) *Gage pressure*. The pressure in excess of atmospheric pressure. Under standard conditions at sea level, the numerical value of the absolute pressure is 14.7 higher than that of the gage pressure. The gage pressure is usually expressed as pounds per square inch gage (p.s.i.g.).

PRESSURIZED FOOD. Food product packaged with a non-toxic compressed gas which serves to self-propel or dispense the food through a suitably valved closure. Food may be dispensed as a spray, foam or as a non-aerated stream.

PROPELLENT. Any non-toxic gas which provides the necessary force to dispense the pressurized food through a suitable valve. Also spelled "propellant."

VALVE. Mechanism for discharging products from pressurized containers. For food products the internal valve orifice must be in the liquid phase. If dispensed with the container upright a dip-tube is required. For inverted dispensing, a "nozzle down" valve without a dip-tube is applicable. A metering valve delivers a definite limited amount of product each time the valve mechanism is operated.

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Packaging Requirements for Pressure Propelled Foods*

(Manuscript received June 3, 1958)

D. W. Riester, R. C. Warner, and H. T. Hoffman, Jr.

Research and Technical Department, American Can Company, Maywood and Barrington, Illinois

WHIPPED CREAM type products have been successfully marketed for a number of years in pressure containers. Their success as well as the great popularity of nonfood aerosols has been due in large part to the functional service performed by the container. For many food products which may be considered for pressure packaging, it is desirable that no extensive change take place in the physical nature of the product during dispensing. The functional service of the container for these products is one of convenience of application, and ease of handling and storage.

Various aspects of the pressure packaging of food products are considered in this paper. Specifically, requirements of the container and associated accessories, propellent, and gassing procedures will be discussed. General observations on other aspects of pressure packed foods are included.

CONTAINER REQUIREMENTS

Container strength. Propellents at the present time are limited to the ordinary compressed gases, such as nitrogen, nitrous oxide, and carbon dioxide. In the pressure ranges acceptable for pressure containers, no liquefied propellent reserve is available as there is in the cases of halocarbon and hydrocarbon type propellents. Therefore, the container must be gassed to a relatively high initial pressure so that an adequate pressure reserve will be available to dispense the entire contents of a can.

These high pressures produce a more severe strain on the container. Body construction must be such that the container will withstand these pressures during extended storage without fatigue failure. Ends must withstand charging pressures without buckling.

The container must also comply with ICC regulations governing the packaging of pressurized products. Regulations may be subject to different interpretations, but it is generally accepted that a pressure not exceeding 105 p.s.i.a. (pounds per square inch absolute) at 70° F. is allowed in a container which is charged with a non-liquefied compressed gas such as nitrogen. A pressure not exceeding 115 p.s.i.a. at 70° F. is allowed in containers packed with foodstuffs and charged with a soluble or emulsified gas such as nitrous oxide or carbon dioxide. In both cases the container must be able to withstand a bursting pressure equal to twice the pressure at 70° F. or one and one half times the pressure at 130° F., whichever is greater.

Corrosion resistance. Conditions exist in a pressure container packed with a food product which are more conducive to corrosion than exist in a conventional tin can of the same product. Headspace corrosion is one of the major problems in pressure propelled products. Headspace corrosion may be more acute in the case of pressure packed products because of the difficulty of minimizing the oxygen content of the final packaged product.

Gas retention requirements. For the successful marketing of pressure propelled foods, containers must be able to retain the initial gas pressure for the expected shelf storage period. Because of the lack of propellent reserve, the container must be essentially gas tight under normal conditions of storage and use.

Years of experience have demonstrated that beer and carbonated beverage containers will retain carbon dioxide at relatively high pressures over long periods of time. However, it was deemed desirable to determine experimentally how well pressure containers will retain compressed gases, nitrogen in particular, at 90 p.s.i.g. (pounds per square inch gauge) at various temperatures. Theoretically, points of possible leakage include the doubleseams of the container, the crimp of the 1 in. cup, and the valve.

An experiment was set up to determine the diffusion rate from the total container. In a second experiment, containers were assembled without valves so that any diffusion rates from the container might be determined independent of the valve. The experiments were thus designed in an effort to evaluate the potential sources of leakage in containers.

EXPERIMENTAL PROCEDURE

Experiment I. Regular three-piece 202 x 406 aerosol containers with one-inch cup openings were fitted with valves of four different manufacturers. Containers were weighed on an analytical balance, and gassed with nitrogen at 90 p.s.i.g. and room temperature; then reweighed and stored at 40° F., 70° F., room temperature, and 131° F.

Weighings were made at monthly intervals and average weight loss values were calculated. Values from 7 to 10 containers were averaged for any one variable.

Weight loss data are presented in Figure 1. Weight loss values rounded to the nearest milligram are plotted against number of months in storage for each valve at each temperature. For containers with valves B, C, and D at the 3 lower temperatures, there appears to have been a small apparent initial weight loss with the rate decreasing with time. However, it is doubtful that the apparent losses are of practical significance because they are in the same order of magnitude as random fluctuations in weighing, which may amount to ± 3 or 4 mg. The average weight losses from containers with valve A were greater at all temperatures.

* Presented at the 18th Annual Meeting of IFT, Chicago, May 27, 1958.

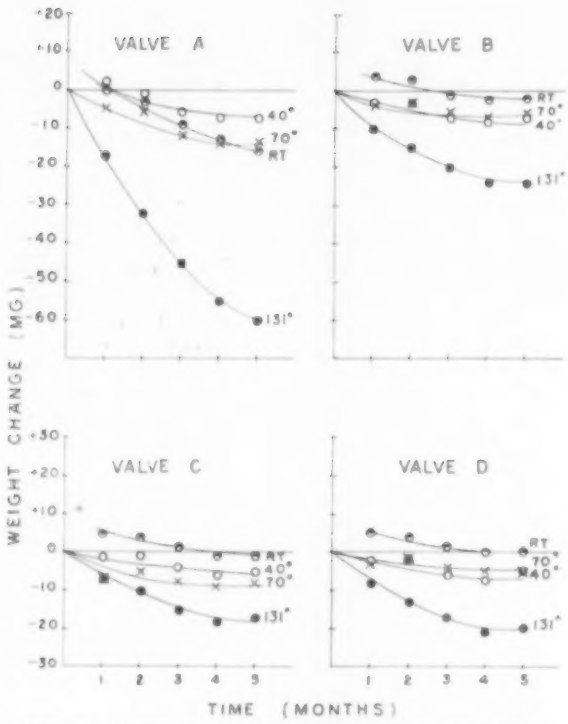

Figure 1. Nitrogen weight loss studies. Time-temperature effect on containers with four different valves.

Greatest weight losses occurred for all variables at 131° F. Containers with valves B, C, and D, have approximately the same weight loss curve. The initial rate loss ranged from 3 to 5 mg./month and appears to have reached a point at 5 months where weight loss is negligible. The initial rate of loss for valve A containers was 15 mg./month. Between the fourth and fifth month, the rate had dropped to 5 mg./month.

Experiment II. In this experiment valveless containers were assembled by double seaming bottom ends on both the top and bottom of aerosol can bodies. Through a hole punched in one of the ends a rubber plug was fitted to provide a means for gassing the container by pushing a hypodermic needle through the plug. On removal of the needle, the rubber plug reseals itself. The details of the experimental procedure were the same as experiment I except that one lot of containers was stored at 98° F. rather than room temperature. Data for this experiment are presented in Table 1 and the values given are the average of 10 to 14 containers. Weight losses from all variables were negligible at the end of 3 months' storage time.

VALVE REQUIREMENTS

Selection of a proper valve is important. The valve and actuator design in combination with propellent will determine to a large extent whether the dispensing characteristics of a food product will be a foam, spray or solid stream.

TABLE 1

Nitrogen weight loss studies. Time-temperature effect on containers without valves

Temperature, ° F.	Weight change		
	1 month	2 months	3 months
40°	+3 mg.	0 mg.	+2 mg.
70°	+3	0	+1
98°	+3	+1	+1
131°	+1	−4	−4

The many kinds of available valves fall into two general groups. The first group is referred to as the foam type valve because it has usually been used for such products as whipped cream. It can also be used for solid stream delivery. Such valves have the advantage of relatively large orifices which permit less restricted flow of fairly viscous products or products with small particles. This type of valve requires that the can be inverted for dispensing and works satisfactorily with products that flow readily toward the valve. However, with more viscous materials, there is danger of loss of gas since the product flows very slowly toward the valve or clings to the wall on inversion of the container. The result is high product retention.

The other group of valves have a dip tube which extends to the bottom of the container permitting dispensing with the can in an upright position. The design of the actuator used on this valve determines to some extent whether the liquid product will be dispensed as a solid stream or as a spray. The smaller orifice size of this type of valve limits its use to relatively fluid products free of discrete particles larger than a few thousandths of an inch in diameter.

The actuator must be non-clogging and non-drip, and must give uninterrupted delivery if a solid stream delivery is desired. For spray dispensing, the actuator must break up the liquid stream mechanically and give an acceptable spray pattern. The small actuator orifices required for spray dispensing must not clog either from drying of product or from excessive particle size.

Many products have a tendency to dry and form a plug in the spout after use. This plug either remains in place during the next usage, in which case it interferes with product flow, or it is dislodged and carried along with the dispensed product, which could be objectionable to the consumer.

Many of the factors mentioned in selection of valves and valve actuators will be determined by the type of product to be packed. Other problems will be resolved only by trial and with the aid of valve manufacturers.

PROPELLENT AND GASSING REQUIREMENTS

Propellents for pressure packed foods have the primary function of forcing the product out of the container. They may also have a secondary purpose of causing the product to foam or to help break it up into a spray.

The selection of a suitable propellent depends primarily upon its ability to deliver a product with the desired characteristics. A product to be dispensed as a foam will require a propellent which is fairly soluble. A soluble gas will also help to some extent in forming a spray because of its tendency to escape from the product when the pressure is released. Solid stream delivery requires a propellent essentially insoluble in the product. Selection of a propellent

should also take into consideration its possible effect on the flavor of the product.

The acceptable gases considered of most importance for use as propellents for foods are nitrogen, nitrous oxide, and carbon dioxide. Nitrous oxide and carbon dioxide are relatively soluble in water and more soluble in fats and oils. Because of this they have found their greatest use in products that are to be dispensed as foams or sprays. Nitrous oxide does have a slightly sweet taste which is not detectable in most food products. Carbon dioxide imparts a slightly acid taste or tang and may be objectionable in some bland products.

Nitrogen is inert to foods and is only very slightly soluble in most food products. It has found its greatest use where it is desirable to have solid stream delivery with a minimum of foaming or bubbling.

Nitrogen propellent studies. As nitrogen became more important as a propellent for solid stream delivery of foods, many conflicting ideas arose concerning various aspects of gassing and gassing procedure. High acid and high sugar products ordinarily preserved by filling and closing hot will also require the same procedure when pressure packed. To insure product sterility, it is therefore necessary to gas while the product is still hot.

A series of tests[1] was initiated in which the effect of agitated versus static gassing and hot versus cold gassing were studied in order to determine: (1) the feasibility of gassing a product hot as related to initial and equilibrium pressures; (2) the weight of nitrogen which could be introduced into a can by different gassing procedures; (3) the effect of gassing procedures and conditions on the appearance of the dispensed product; and (4) the residual pressure when all product is discharged from the can.

Test procedures. The tests were conducted with water, 30° Brix syrup, 60° Brix syrup, chocolate syrup, catsup, and cottonseed oil. All tests were made with 211 x 604 pressure cans having a one-inch cup opening and a fill of 11 fluid ounces. The fill was determined by the weight of 11 fluid ounces of each product at room temperature.

In cold fill tests, 24 cans of each product were filled and closed at room temperature. They were then weighed to within 4 or 5 mg. on an analytical balance which had a capacity of 750 g. Twelve cans were then gassed with agitation and 12 cans were gassed statically with nitrogen at 90 p.s.i.g. They were reweighed and the weight of nitrogen introduced into the cans determined by difference in weight before and after gassing. The agitated samples were gassed on a shaker[a] operating at 350 cycles per minute.

In tests with hot fill and gassing, each product was filled and closed at 190° F. Cans were allowed to cool during weighing. They were then reheated to 190° F. and gassed at 120 p.s.i.g. with nitrogen. Agitated and static gassing were used with each product. Weight of nitrogen was again determined by difference.

The pressures measured after 24 hours were considered to be equilibrium pressures. Pressures were also obtained on cans of each product after extended storage. All pressures were taken with a prepressurized gauge.

Weight of nitrogen in can. Table 2 shows the weight of nitrogen introduced by different gassing procedures, equilibrium pressures, and residual pressures.

Close agreement was found between the experimental and calculated weight of nitrogen which could be introduced into a 16-ounce pressure can filled with 11 fluid ounces and gassed statically at 90 p.s.i.g. at room temperature. A 211 x 604 pressure can has a capacity of about 17.5 fluid ounces which leaves a 6.4 fluid ounce headspace when filled with 11 fluid ounces. Theoretically, the weight of nitrogen which can be introduced into this space under the conditions mentioned, assuming air at one atmosphere pressure already present in the headspace, would be

[a] Alpha Engineering and Machine Works, Inc., Mount Prospect, Illinois.

TABLE 2

Nitrogen propellent studies. Comparison of gassing procedures

Product[1]	Gassing temp. (° F.)	Gassing press. p.s.i.g.	Static gassing				Agitated gassing			
			Wt. N_2 (g.)	Equilibrium press. (p.s.i.g.)		Residual press. after dispensing (p.s.i.g.)	Wt. N_2 (g.)	Equilibrium press. (p.s.i.g.)		Residual press. after dispensing
				24 hrs. at R.T.	1–2 mos. at 70° F.[2]			24 hrs. at R.T.	1–2 mos. at 70° F.	
Water	RT	90	1.350	86	84	26	1.417	90	88	28
Water	190	120	1.460	82	80	24	1.484	84	82	25
30° Brix Syrup	RT	90	1.343	86	83	27	1.410	89	87	28
30° Brix Syrup	190	120	1.434	83	80	24	1.471	85	82	25
60° Brix Syrup	RT	90	1.368	88	87	28	1.421	90	90	29
60° Brix Syrup	190	120	1.437	87	85	27	1.456	86	84	27
Chocolate Syrup	RT	90	1.333	88	88	28	1.356	89	88	28
Chocolate Syrup	190	120	1.464	88	88	29	1.453	85	84	27
Catsup	RT	90	1.335	87	85	33	1.382	90	88	35
Catsup	190	120	1.431	84	80	30	1.405	84	81	30
Oil	RT	90	1.339	85	78	27	1.468	87	81	28

[1] Fill, 11 fl. oz.
[2] Cold filled products stored about 2 months. Hot filled products stored about one month.

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about 1.335 g. Experimentally the average weight introduced into cans of all products ranged from 1.333 to 1.368 g.

Agitation of most products filled and gassed cold showed only slight increase in the weight of nitrogen which could be introduced into the cans. Agitation resulted in an average increase of 2 to 5% for all products. Oil was a notable exception. It showed a substantial increase of 9% in the amount of nitrogen that could be introduced by agitation. This is attributed to the fact that nitrogen has a greater solubility in oil.

Table 2 shows that more nitrogen by weight is required to gas hot filled cans than cold filled cans to arrive at the same equilibrium pressure at room temperature. The reason is that the headspace of the cold filled cans contained one atmosphere of air when closed, whereas the headspace of the hot filled cans was partially occupied by product vapors.

Hot filled products gassed statically required 5 to 8% more nitrogen than cold filled products gassed statically. With agitated hot filled products, the weight increase was 2 to 7% over cold filled cans gassed with agitation. In general, agitation had little or no effect on the weight of nitrogen introduced into cans gassed with the product at 190° F.

Feasibility of hot gassing. Results of these tests indicate that charging pressures necessary to gas a product with nitrogen at 190° F. are not excessive. Charging pressures required are 30 to 40 p.s.i.g. higher than the pressure desired at 70° F. The exact charging pressure will have to be determined by trial, however, since it will depend to some extent on the vapor pressure of the product.

The effect of vapor pressure on equilibrium pressure is shown by the different hot gassed products. Water with a high vapor pressure showed a greater pressure drop than syrups which have lower vapor pressures.

Residual pressures. Pressures of 85 to 90 p.s.i.g. were sufficient to dispense the 11 fluid ounces of each product tested. With less fill, even smaller initial pressures may be adequate.

The calculated pressure which should remain when 11 fluid ounces of product are completely discharged from a 16-ounce can having an initial pressure of 90 p.s.i.g. would be 23 p.s.i.g. Residual pressures in cans of each product tested ranged from 3 to 12 p.s.i.g. greater than this depending on the retention of product in the can. In no instance was all of the product dispensed.

More fluid products such as water and 30° Brix syrup showed residual pressures 4 to 5 p.s.i.g. greater than calculated with a 3 to 5% product retention. Catsup which had 25 to 30% retention showed residual pressures 10 to 12 p.s.i.g. greater than the calculated pressure which should remain if all the product was discharged.

DISPENSING TESTS

Product appearance. The following observations on dispensing were made within a week after packing.

Even though nitrogen is only slightly soluble in most food products, this slight solubility may have an objectionable effect on the dispensed product. The method of gassing, agitated or static, also has an effect on the characteristics of the dispensed product. Hot or cold gassing shows no difference in effect on the dispensed product.

Water and 30° Brix syrup, gassed either with agitation or statically, were dispensed with a milky white appearance due to small gas bubbles. The bubbles dispersed quickly, however, and may or may not be considered objectionable.

When the more viscous chocolate and 60° Brix syrup were gassed with agitation, sufficient gas was trapped in the syrups to make them have a foamy appearance when dispensed. When these products were gassed statically, the product dispensed satisfactorily although some bubble formation was noticed eventually beneath the surface of the clear 60° Brix syrup.

Catsup was dispensed with only very slight discoloration when it was gassed in a static condition. When it was gassed with agitation it dispensed with an objectionable orange color. It is not known whether this effect would be noticed after extended storage.

Nitrogen is somewhat more soluble in oil than in other products. This solubility caused considerable frothing which continued for 1 to 2 minutes.

Product retention. Another problem facing the pressure packing food field is that of excessive product retention in the case of higher viscosity products. Cavitation may also occur with some products on dispensing, allowing the gas to escape and leaving large quantities of product hanging on the container walls.

To date this problem has been alleviated by product reformulation to a lower viscosity. Treating the container walls with a slip compound and/or a lubricating oil to reduce product clingage to the side walls possibly may be helpful with some products.

Dip tube type valves require indexing the valve cup so that the actuator can be aligned with the dip tube to insure adequate removal of the product. The natural curvature of the dip tube allows the lower end of the tube to be positioned at the sidewall of the lower end of the container. By locating the actuator spout on the same side of the container, the dip tube is at the lowest point in the can when it is tilted forward.

Product stability. Pressure packing itself has no inhibiting effect on the growth of spoilage organisms. Essentially the same rules should apply to pressure packing as for regular packing *(1)*. High acid and high sugar products ordinarily preserved by packing hot should be filled and gassed hot in order to prevent spoilage. It was shown earlier that this packing procedure was feasible for pressure packing. Pressure packing of low acid foods which ordinarily require processing at 240° F. or higher must be considered an entirely different problem. Special aseptic canning procedures requiring complex equipment and precise control must be used. Adaption of aseptic

canning to pressurized foods presents formidable problems.

Product changes that occur upon dispensing in which gas solubility is not a factor are also sometimes observed. Products with a gel structure may have a tendency to break down under pressure or on dispensing; those with thixotropic properties may set up after standing, but immediately after dispensing give the appearance of a reduced viscosity material.

Color and flavor retention are other factors which must be considered. Test packs which have been stored for extended periods enable selection of container and valve components which are compatible with the product. All materials which come in contact with food should be acceptable to the Food and Drug Administration.

SUMMARY

General packaging requirements as well as problems connected with pressure packaging of food products have been discussed in this paper. An experimental study of gassing and filling procedures was presented. This study demonstrated the feasibility of hot filling and gassing. An experimental determination of the loss of nitrogen from pressure containers has shown the loss to be negligible under ordinary conditions for the period of several months that the test has been under way.

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Heat Preservation of Pressure Dispensed Food Products*

E. D. Giggard and P. B. Gottschall

Metal Division Research and Development Department, Continental Can Company, Inc., Chicago 20, Illinois

(Manuscript received May 11, 1958)

Pressure dispensed foods are of particular interest to food technologists in that they present many challenging problems unique to this type of packaging. Results of work on some of the basic problems involving product dispensing characteristics, gas propellents or propellent mixtures, propellent solubility, temperature-pressure relationships, and container and valve designs have been reported. *(3, 9)*. However, the problem of attaining biological stability and certain other technological problems, such as flavor and color retention and flow chacteristics peculiar to specific types of products, must be resolved before it will be feasible to package a wide variety of foods in pressure dispensing containers. Until recently, whipped cream and synthetic whipped toppings were the only pressure dispensed foods generally available to the consumer market. The majority of these products are merchandised under refrigeration, with a resultant product shelflife of six to ten weeks. However, refrigerated handling is impractical for the majority of food items being considered for pressure dispensing for several reasons:

1. The rheological properties of many products are altered at refrigerated storage temperatures to such an extent that the product cannot be pressure dispensed satisfactorily at practical pressures;

2. Economic disadvantage in shipping, storage, and marketing;

3. Inadequate propellent pressure at lower temperatures; and

4. Product shelflife limited to less than 3 months for many products.

Food manufacturers are showing intensive interest in merchandising a wide variety of fluid and semi-fluid products such as liquid coffee concentrate and peanut butter in pressure dispensed form. The impracticability of merchandising most pressure dispensed foods under refrigeration makes it imperative that the subject of preservation of these products via other means be considered at this time. Of the several available means of preventing or inhibiting microbiological spoilage under study at present, the following appear to have greatest application to pressure dispensed foods:

1. Direct application of heat before or after filling;

* Presented at the Eighteenth Annual Meeting of the Institute of Food Technologists, Chicago, Illinois, May 27, 1958.

2. Control of product composition: pH, salt content, moisture content, etc.;
3. Maintaining aseptic packing conditions;
4. Refrigeration for special classes of products, despite some disadvantages;
5. Use of chemical preservatives;
6. Use of high frequency electrical energy: dieletric, induction or resistance heating; and
7. Use of ionizing radiations.

The suitability and practicability of these methods depend, in varying degrees, upon product composition and packing procedure for their effectiveness. Adaptation of the latter two principles to food preservation is presently in the development stage; they are mentioned, however, merely as possibilities for the future.

To date, experience in packing pressure dispensed foods has been limited to products such as barbecue sauce and table syrups, in which commercial sterility can be achieved at temperatures below 212° F. by employing a hot fill. Methods of preserving these products in pressure dispensed form differ from those used for conventional, hermetically sealed containers in that after the product and container are heat treated, the valve is opened to permit charging the gas propellent into the container, thus presenting a potential spoilage hazard through recontamination of the product. Therefore, studies were initiated to evaluate various heat preservation techniques, and to determine their practicability and adaptability to foods packaged in pressure dispensing containers. It is our prime purpose to report the results of these investigations; to discuss means whereby heat can be used to preserve various types of pressure dispensed foods against microbiological spoilage; and to point out the advantages and limitations inherent in the various methods investigated. It is our hope that this information will be of value in stimulating further interest in research on the heat preservation of pressure dispensed food products.

EXPERIMENTAL PROCEDURE

All pressure studies reported were conducted using commercial three-piece 12-ounce aerosol cans, except as noted. One series of tests was designed to determine equilibrium pressures attained by charging compressed gases directly into hot products. In this experiment various products were filled, 6-, 8-, and 10-fluid ounces at temperatures of 140° F., 160° F., and 180° F., immediately vacuum crimped and charged. Propellents used include nitrous oxide (N_2O), carbon dioxide (CO_2), N_2O (85%) + CO_2 (15%), or nitrogen gas (N_2) at pressures ranging from 100 to 150 p.s.i.g. in 10 pound increments. All cans were water tested after charging to make certain that there was no gas leakage. After 24 hours storage at 70° F. each container was shaken for 15 seconds on an agitating charger and its internal pressure determined by means of a gauge system pre-pressurized to the anticipated pressure.

Rate of temperature change during cooling in similarly packed containers was next determined by means of a recording potentiometer and copper-constantan thermocouples located in both the product and propellent phases of the container. Data were obtained both with and without agitation of cans during the cooling period. Rate of product and gas temperature change while the cans were submerged in a water bath was determined in the same manner.

Relationship between internal pressure and temperature was determined by packing containers equipped with a thermocouple in the product area and a pressure gauge connected to the headspace area. Temperature changes were recorded from a potentiometer, and pressure readings were observed simultaneously from the pressure gauge as the container was agitated vigorously in a hot (190–200° F.) water bath.

Commercial valves of several types and manufacture were tested to ascertain their capacity to maintain a high internal vacuum for a period of 5 minutes. This was accomplished by incorporating individual valves into a system containing a closed tube mercury manometer, and to which a vacuum pump was attached. A glass stopcock was placed between the valve to be tested and the vacuum pump. In operation, pressure in the entire system was reduced to 1 mm. mercury pressure after which the test portion was sealed-off by means of the stopcock. Vacuum loss was read on the manometer at intervals of 1, 3, and 5 minutes.

In order to determine the possibility of pressure processing filled containers charged with compressed gas, cans were filled with 9-fluid ounces of water and charged with 90 p.s.i.g. N_2O pressure or 100 p.s.i.g. N_2 pressure and heat processed. The N_2O charged cans were processed (50 cans each) in a conventional retort and in a glass retort installation with 40 p.s.i.g. air overpressure for 30 minutes at 240° F. The N_2 charged cans were processed in a conventional retort as follows: 100 cans processed 30 minutes at 240° F., and 50 cans processed 30 minutes at 250° F.

Cooling at completion of the process was accomplished by releasing the steam pressure slowly, followed by water cooling to 100° F. center temperature. Cooled cans were examined for leakage and end distortion, and stored at 130° F. All cans in 130° F. storage were examined semi-weekly for a period of one month for evidences of container stress failure.

RESULTS AND DISCUSSION

Products requiring only mild heat treatment. Certain types of food products subject to spoilage only by non-spore forming organisms may be preserved by mild heat treatment. Such products would be represented by those having a pH below 4.0, such as salad dressings, or a sufficiently high concentration of dissolved sugar or sodium chloride, such as honey or beef extract, to prevent the outgrowth of surviving spores. If it were not for the problem of possible recontamination of the product during subsequent charging with propellent, the procedure of filling the product hot, sealing, and inverting the container for several minutes at a product temperature of 170–200° F. would provide positive commercial sterility for these products. However, gas charging the product while still hot or after cooling is a necessary step in the procedure that must be considered.

Charging Cold Product Under Semi-aseptic Conditions. If the commercially sterile product and container are obtained by means of the hot fill-hold and cool technique, the problem of maintaining sterility becomes one of maintaining aseptic conditions while charging propellent into the cooled container. Propellent gas can be effectively rendered sterile by filtration. However, there remains the possibility of forcing contaminate trapped on the interior valve stem surfaces into the container along with the propellent. The area of possible contamination in this type of operation extends from the cooling equipment where a partial vacuum develops in the container, through the charging operation where a high positive pressure is secured within the container. However, we believe that with proper precaution

spoilage in this type of operation can be eliminated. Recommended precautionary measures against contamination are as follows:

1. Control the bacteria count of cooling water by chlorination *(4, 6, 8)*, maintaining the level of residual chlorine in the equipment at approximately 2–3 p.p.m. *(2)*. Work with a variety of valve styles has shown that several valves are very efficient in maintaining a vacuum of 1 mm. mercury pressure within a container for 5 minutes. Similar valves of different manufacture are not, however, equally efficient in this respect. During the cooling process, the development of a vacuum in the can could result in the aspiration of minute amounts of cooling water into the can. Thus it is imperative that the bacterial content of the cooling water be maintained at the lowest possible level, to avoid spoilage from contaminated cooling water *(6)*.

2. Cans leaving the cooling apparatus trap varying amounts of cooling water in the outer portion of the valve stem depending upon valve design. It is recommended that a high velocity, air or steam jet be directed downward upon the valve to remove this water so that it is not forced into the container during the charging operation.

3. Valve stems are subject to contamination by airborne organisms while being conveyed from the cooling operation to the charging operation. It is recommended that a bank of ultraviolet lamps, producing a high output near 2,600 Å *(1)*, or a tunnel of live steam be employed to reduce the possibility of contamination at this point.

Charging Hot Product. Since recontamination during charging is a problem, the possibilities of charging the propellent directly into the hot product —thereby transferring heat from the product to both container and propellent—were investigated. This procedure necessitated major upward adjustments in charging pressure due to decreased dissolution of the propellent in product, decreased density of the propellent, and decreased headspace volume at elevated temperatures. The relationships between propellent charging pressure and equilibrium pressure (at 70° F.) with different products, propellent gases, volumes of fill, and product temperatures are illustrated in Figures 1 and 2. It is apparent that regardless of product and propellent gas employed, equilibrium pressure (at 70° F.) resulting from a given charging pressure varies inversely with both product fill and product temperature at time of charging. A linear relationship exists between charging pressure and equilibrium pressure with all compressed gas propellents studied. A reasonably accurate prediction of equilibrium pressure conditions resulting from a wide range of products, product fill ratios, propellents, and product temperatures from a single pressure determination is therefore possible.

The effect of propellent solubility on equilibrium pressure (70° F.) in hot charged products is illustrated in Figure 1: nitrogen representing a relatively insoluble propellent and carbon dioxide a relatively soluble propellent. Comparison of carbon dioxide charging data presented in Figure 1, and nitrous

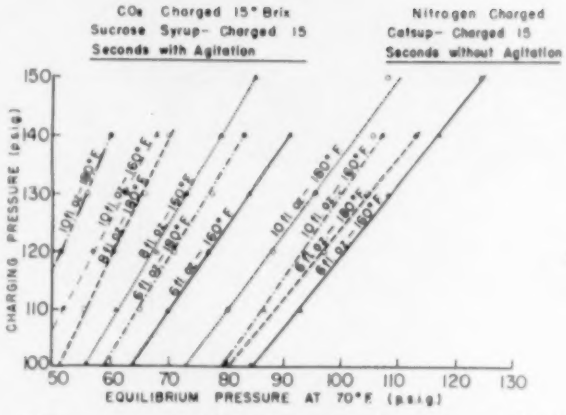
CO₂ Charged 15° Brix Sucrose Syrup– Charged 15 Seconds with Agitation
Nitrogen Charged Catsup– Charged 15 Seconds without Agitation
CHARGING PRESSURE (p.s.i.g)
EQUILIBRIUM PRESSURE AT 70°F (p.s.i.g)

Figure 1. Effect of product fill, product temperature at time of charging, and charging pressure on equilibrium pressure (70° F.) of two food products.

oxide (85%) + carbon dioxide (15%) data from Figure 2, reveals that pressure characteristics of the two propellents in two different products are quite similar.

Heating and cooling rates of compressed nitrogen when charged into a hot product and corresponding temperature change in the product are illustrated in Figure 3. The high rate of thermal conductivity of

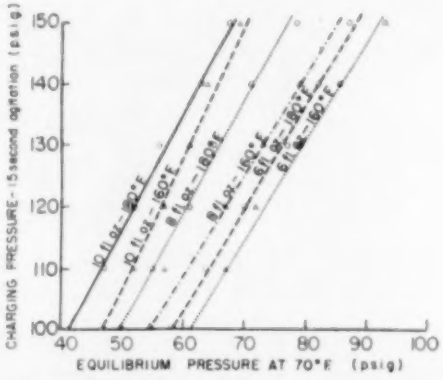

Figure 2. Effect of product fill, product temperature at time of charging, and charging pressure on equilibrium pressure (70° F.) of N_2O (85%) + CO_2 (15%) charged corn oil.

compressed gases is apparent in both heating and cooling rate curves. Achieving any practical degree of lethality in the propellent proper would therefore require an auxiliary means of maintaining propellent temperature near the product temperature for several minutes. A hot water bath or continuous oven was shown to be very effective in rapidly heating and maintaining the temperature of the gas at the required level. Agitation during the charging operation has been shown to be very effective in maintaining propellent temperature at the product temperature. However, the shaking cycle is relatively

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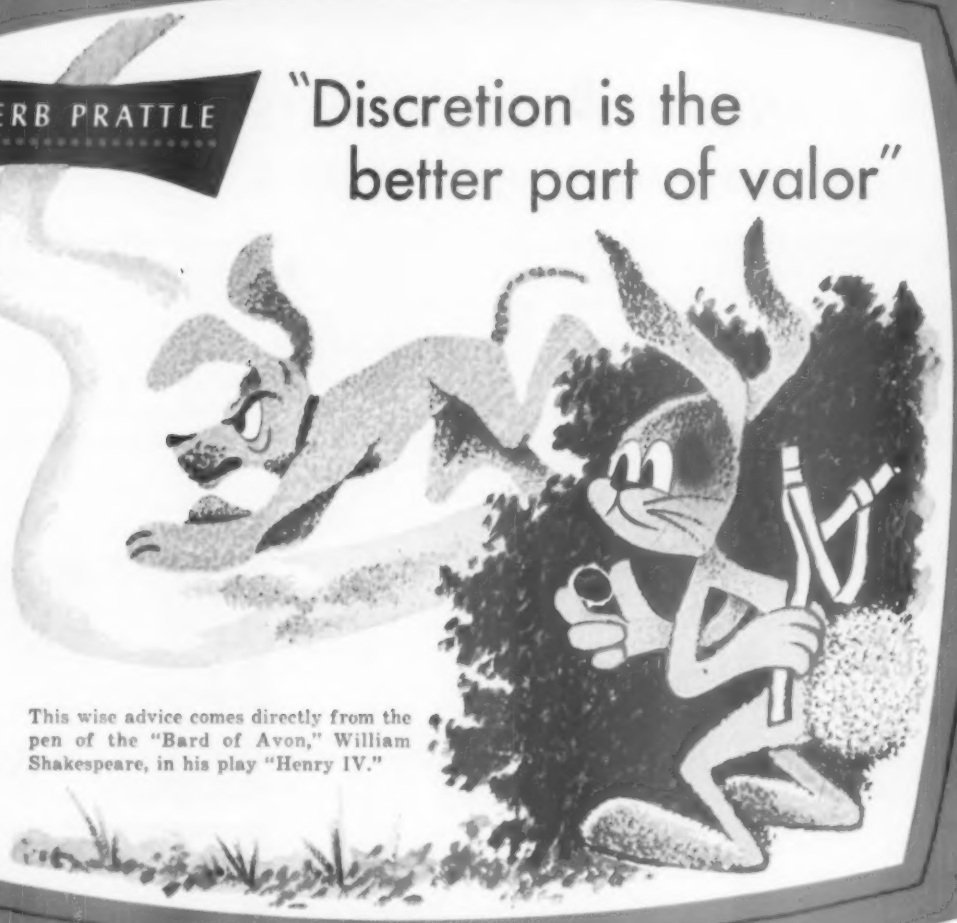

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HOFFMANN-LA ROCHE INC.
Nutley 10, N. J. • NUtley 2-5000
In New York City: OXford 5-1400

In Canada: Hoffmann-La Roche Ltd., 1956 Bourdon Street, St. Laurent, Montreal 9, P. Q.

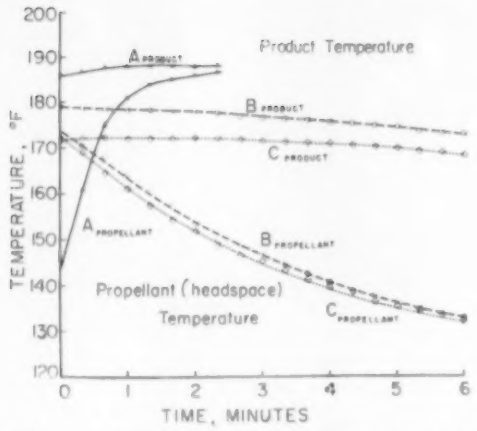


Figure 3. Rate of temperature change in food products "hot charged" with N_2. (A = Catsup immersed in 190° F. water bath; B = Chocolate syrup cooled in air at 82° F.—no dip tube on valve; C = Chocolate syrup cooled in air at 82° F.—dip tube on valve.)

short (10–20 sec.) and when agitation ceases propellent temperature again decreases rapidly.

Data presented in Table 1 also show that agitation is not essential in packaging with nitrogen as the propellent.

Charging hot (180–200° F.) products with soluble gases such as N_2O and CO_2 is impractical as required charging pressures are excessive. For example, a charging pressure of approximately 175 p.s.i.g. is required to produce an equilibrium pressure (70° F.) of 90 p.s.i.g., in a 12 fl. ounce container filled with 8 fl. oz. of 15° Brix syrup at 180° F. and charged with CO_2.

TABLE 1

Effect of agitation during charging (70° F.) on equilibrium pressure (70° F.) of nitrogen charged chocolate syrup

Sample	Charged 90 p.s.i.g. N_2—No agitation		Charged 90 p.s.i.g. N_2—20 sec. agitation	
	Weight of N_2 added in grams	Residual pressure after storage 24 hrs. @ 70° F.	Weight of N_2 added in grams	Residual pressure after storage 24 hrs. @ 70° F.
1	1.087	88 p.s.i.g.	1.135	91 p.s.i.g.
2	1.105	89 p.s.i.g.	1.140	90 p.s.i.g.
3	1.120	88 p.s.i.g.	1.175	91 p.s.i.g.
4	1.080	88 p.s.i.g.	1.150	91 p.s.i.g.
5	1.128	88 p.s.i.g.	1.175	92 p.s.i.g.
6	1.133	88 p.s.i.g.	1.195	90 p.s.i.g.
Avg.	1.109	88.2 p.s.i.g.	1.162	91.0 p.s.i.g.

Several advantages and disadvantages of the above method of achieving product sterility are apparent:

Advantages:

1. The majority of the vegetative organisms which might be introduced during charging with propellent would be destroyed by the heat of the product.

2. When employing the hot charge procedure, a vacuum would not be formed within the can during the cooling period, thereby eliminating the potential hazard of drawing contaminated cooling water through the valve into the product.

3. Where applicable, this means of heat treatment would not decrease the normal production rate or require additional packaging equipment.

Disadvantages:

1. Higher charging pressures are required to attain the desired equilibrium pressure at 70° F. The higher charging pressures would place additional temporary stress on container and valve components, and would also magnify small pressure irregularities in containers charged on different stations of multihead charging equipment.

2. This method would not be applicable when a high degree of solubility of propellent in product occurs.

Based on the above data, it appears both possible and practical to achieve commercial sterility in acid products or products containing high sugar or salt content by charging relatively insoluble gases such as nitrogen directly into the hot product, and holding at an elevated temperature followed by rapid cooling to 80–90° F.

Products requiring high heat treatment. The preceding techniques offer possible means of preventing spoilage in products incapable of supporting growth of organisms which constitute a potential health hazard. However, in dealing with food products in which product composition is not effective in inhibiting growth of heat tolerant microorganisms, the heat treatment provided by these methods is entirely inadequate to render the product commercially sterile.

Precluding the possibility of merchandising under refrigerated conditions, available means of preserving liquid products of this type include processing or heat treating the completed package, aseptic canning, or use of chemical preservatives. Since our investigations were confined to heat preservation techniques, the use of chemical preservatives will not be discussed at this time.

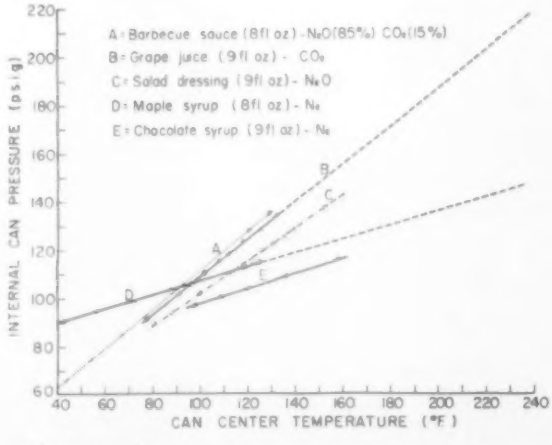


Figure 4. Effect of can center temperature on container equilibrium pressure.

PRESSURE PROCESSING

It was originally assumed that processing pressurized containers would be impractical due to the high internal pressures developed at elevated temperatures. Pressures actually encountered with various products and gas propellents over a portion of the temperature scale and projected into the processing temperature range are shown in Figure 4. Data illustrated in this figure show that:

1. Linear relationships exist between temperature and internal container pressure for all products and gas propellents studied.

2. The pressure curve for products charged with soluble propellents, i.e. CO_2, N_2O and $CO_2 + N_2O$ is steeper (approximately 7 p.s.i.g./10° F.) than that for N_2-charged products (approximately 3 p.s.i.g./10° F.). This difference is due primarily to decreased solubility of the more soluble gases at elevated temperatures.

3. Pressure curves of all the soluble propellent gases are quite similar.

Present specification commercial aerosol containers of both 202 and 211 diameters are incapable of withstanding the pressure (approximately 218 p.s.i.g.) developed within a food product, when charged with soluble gases of 90 p.s.i.g. (70° F.) and processed at 240° F. The major incidence of failure under these conditions is due to end unit buckling. Buckling of container end units at this processing temperature was not relieved either by processing under 40 p.s.i.g. total pressure (10.3 p.s.i.g. steam pressure + 29.7 p.s.i.g. air pressure) or by pressure cooling the container at completion of the process. Data available indicate that present 202 diameter container designs could be made to withstand the above process by increasing the thickness (base weight) of the plate used for end units. Adaptation of the 211 diameter container to meet the same requirements would probably require both a modification of the structural design of the end units and an increase in base weight of end unit plate.

Pressure processing of nitrogen-charged 211 diameter cans, however, appears to be feasible. Commercial specification 12 fl. oz. aerosol cans were filled with 9 fl. oz. of water and charged with nitrogen to 100 p.s.i.g. pressure. No permanent can distortion or evidence of leakage was observed in 100 of these containers processed for 30 minutes at 240° F. or in 50 containers processed for 30 minutes at 250° F. None of the above cans were pressure cooled. Estimated pressures developed within the containers during pressure processing were 149 p.s.i.g. at 240° F., and 152 p.s.i.g. at 250° F. Semi-weekly examination of the 150 processed cans and 100 nonprocessed cans for a period of one month, under conditions of 118 p.s.i.g. internal pressure at 130° F., showed that the pressure process had no effect upon subsequent container performance.

Pressure processing places abnormal demands upon valve components as well as container components. Acceptable plastic materials must possess a heat distortion temperature in excess of 240° F. at 150 p.s.i.g. pressure, and a minimum degree of dimension change under similar conditions. Of the present plastic materials used in aerosol valve manufacture, i.e. polystyrene, polyethylene and nylon, only nylon approaches satisfactory performance under the above conditions. It is possible that by utilizing the proper filler material polystyrene can be made sufficiently heat resistant for limited use. The main uses of polyethylene in valve manufacture are for dip tubes and actuators. Actuators need not be applied until after cans are heat processed and cooled. Since no particular stress is imparted to the dip tubes, high modulus type or radiated *(5)* polyethylene should provide sufficient heat resistance for use in these parts.

Other plastic materials which may prove useful in manufacturing valves for use on heat processed products include melamine formaldehyde resins, phenol formaldehyde resins, silicone compounds and isotactic polypropylene *(7)*.

Advantages of sterilizing pressure dispensed foods by pressure processing are obvious:

1. Sterilizing values necessary to preserve different products are easily attainable by regulating the processing time.

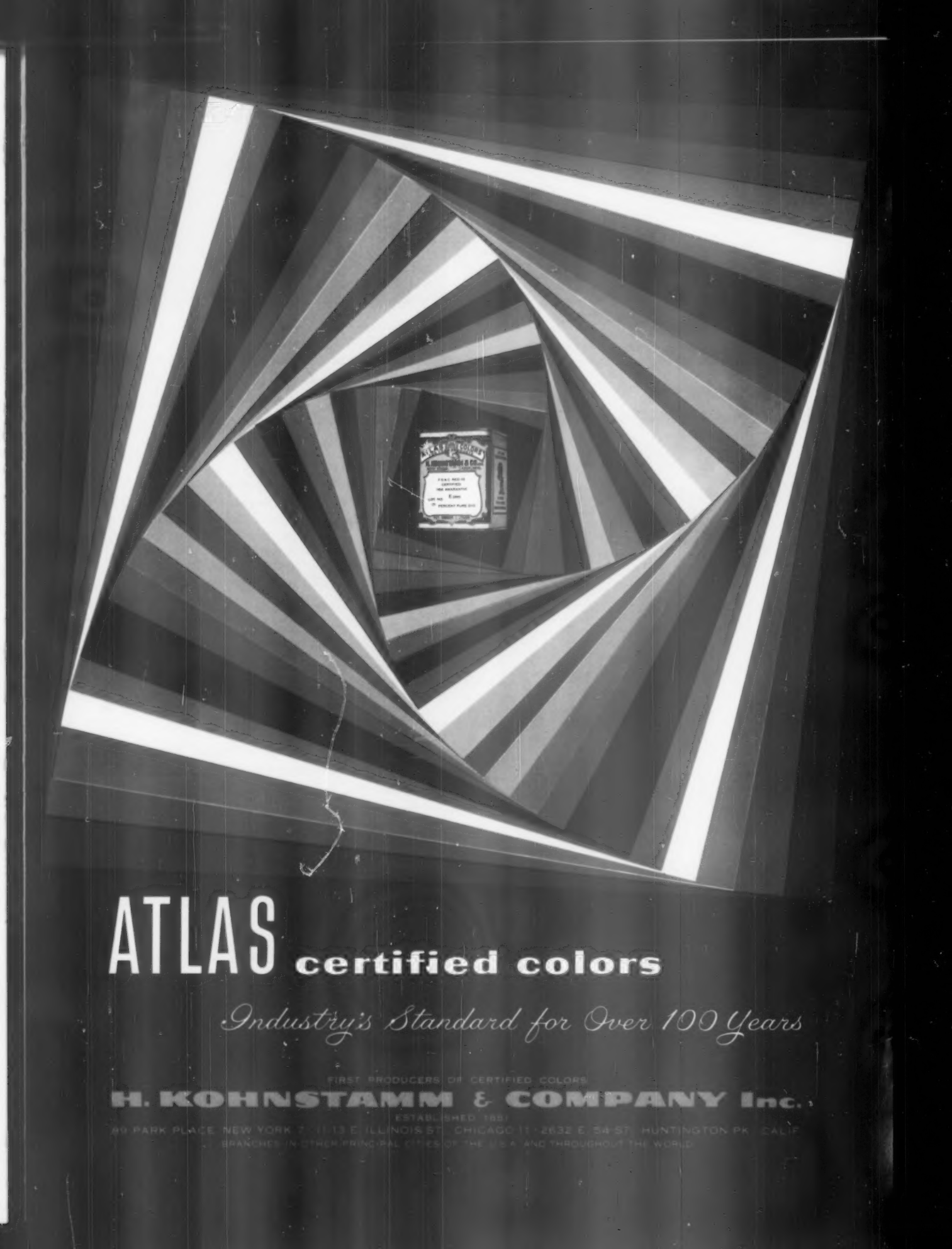
2. Commercial sterility is attained with the package in its final form; therefore, there is no opportunity for recontamination of the product.

The major disadvantage of pressure processing stems from the great physical stress placed on the container and valve components during the processing period, demanding a container with unusual performance characteristics. However, as stated previously, the problem can be overcome by judicious use of container materials, designs, and structures at probably a somewhat greater cost than today's conventional container.

ASEPTIC CANNING

Commercial aseptic canning of a pressure dispensed whipped cream topping by the "Avoset Process" has been successfully employed by the Avoset Company, San Francisco, California, since 1950. Several systems of aseptically packing have been patented, i.e., the Martin Process, Avoset Process, HCF Process, and the Smith-Ball Process. While the mechanics of the processes differ, they are the same in principle. All involve heat sterilizing the product, cooling, and subsequently filling and sealing the sterile product in presterilized containers under aseptic conditions.

The major advantage inherent in this type of processing is that the product is sterilized in a heat exchanger under ideal conditions providing rapid and thorough heating of the product. Rapid heating permits sterilization of some heat sensitive products which would be rendered unmerchantable by conventional in-can processing methods. The method is equally effective in preserving less heat sensitive products. Aseptic canning provides a method of achieving commercial sterility in low acid products without subjecting containers to the high pressures involved in pressure processed products. This is the



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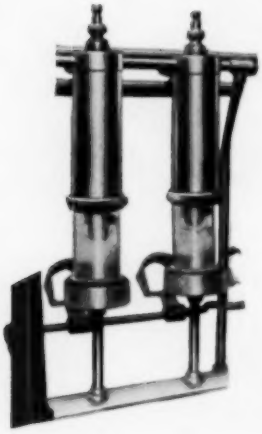

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only method which appears to be immediately adaptable to processing low acid food products charged with soluble gas propellents.

The major disadvantage of aseptic canning is the relatively expensive and elaborate equipment requirements. In addition, present commercial aerosol valves containing plastic components would not withstand the heat treatment employed to produce sterile containers and closures for filling.

SUMMARY

Methods are available for achieving commercial sterility in pressure dispensed food products. The procedure used, and ease of attaining commercial sterility, however, depend largely upon the physical and chemical nature of the product in question.

For products such as barbecue sauce, salad oil, or sugar syrups in which the acid content or osmotic pressure is sufficient to prevent the growth of spoilage organisms and/or toxin production by heat tolerant organisms, mild or no heat treatment is sufficient to achieve commercial sterility. Several methods of achieving a mild heat treatment in order of increasing sterilizing effectiveness include hot filling, hot charging, and pasteurization.

Products such as vegetable purees and meat spreads are capable of supporting growth of toxin producing organisms and must be given a high heat treatment in order to eliminate potential health hazards and to destroy heat resistant spoilage organisms. Commercial sterility in this group of products may be achieved either by pressure processing, if the product is charged with nitrogen or argon, or aseptic packaging. Where pressure processing is to be employed, special consideration must be given to selecting container materials capable of withstanding the rigorous pressure and temperature conditions inherent to the method.

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How a Food Manufacturer Can Start and Complete a Pressurized Food Program*

(Manuscript received May 11, 1958)

R. C. Webster
Ohio Chemical & Surgical Equipment Co., Madison, Wisconsin

THE PRESSURIZED FOOD FIELD, as we know it today, is nearly 10 years old; yet it is perhaps the newest facet in an evergrowing convenience-food market. In the past year new true convenience "push button foods" and related items, other than whipped creams, have appeared on the market. Among these are a spray barbecue sauce *(4, 7, 8)*, a non-aerated chocolate syrup *(2)*, and now several different brands of non-aerated toothpaste. Other products that are in or near the marketing stage are salad dressings, cake icings, meat tenderizers, a multitude of barbecue sauces, and many, many others *(1, 6)*. The interest created by the advent of these few products has been phenomenal. Many firms are now working feverishly to get new products on the market, thus creating a "first." One of the reasons why more new products have not appeared, each in their own light a "first," has been the lack of properly guided development programs.

There are many questions that management and research are concerned with during preliminary discussions on application of their products to the pressure package. Among these are economics, packaging problems, and marketability of the potential product. Upon answering these questions, the next question is usually, "How can we start a pressurized food program and carry it to a profitable conclusion?"

The purpose of this paper is to point out various factors that may ultimately prove to be of assistance in establishing a profitable program, a program profitable not only from the standpoint of economics, but one which yields a product of the highest quality and embodies the true features of convenience and utility.

ESTABLISHING A PROGRAM

Evaluation of product. The primary questions that must be answered prior to the inception of a research program on pressurized foods are numerous. Some of these are:

1. Do we have a suitable product for this type of package, and does it meet standards of convenience?
2. What market potential has this product in the pressurized form?
3. What are the component cost factors and how do they relate to merchandizing?
4. Where can we go for specialized assistance?
5. What are the governmental regulations involved in the packaging and merchandizing of a product?
6. Should we install packaging facilities or have the product packaged by a custom packager?
7. What is the capital investment involved in a venture of this sort?

Each of the above factors is of great significance and relates to each and all of the other factors.

The pressurized food, if it is to be successful, must have inherent quality and convenience. Webster defines convenience as "any labor-saving or comfort giving appliance, or fixture, etc." If the pressurized food saves the housewife time and labor, and yet offers a useful and worthwhile application, it will be classed in the consumers' mind as a convenience item. If the product is of high quality and is competitive costwise as well, it will become a practical everyday convenience item rather than a luxury.

The ultimate market potential of a pressurized food product is a question much in the minds of all manufacturers. It has been proven that the American consumer will pay extra for convenience, and continue to pay for convenience when it is combined with quality. If these two factors are present, the success of the product then rests with the ability of the company to promote and sell their product.

The cost of a product in a pressurized container will in most cases be higher in unit price as compared to the same product in a glass or plastic container. The increase in price structure is due to a slightly increased container cost, as well as the cost of other fitments such as valve, actuator, overcap, etc. A comparison in the costs of a 12 fluid ounce glass bottle of barbecue sauce versus the cost of a 12 fluid ounce fill of pressurized barbecue sauce is shown in Table 1.

TABLE 1

Relative cost comparison (approximate) of barbecue sauce (12 fluid ounce fill) in glass versus a 12 fluid ounce fill in a pressure can

Component	Metal container	Glass container
	(Price per thousand)	*(Price per thousand)*
Package	$ 60.00	$ 50.00
Product	120.00	120.00
Valve	33.00	.....
Screw Cap (Bottle)	.....	10.00
Actuator	10.00	.....
Overcap	15.00	.....
Label	7.00	5.00
Total price	$245.00	$185.00

[1] Prices above do not show filling charges, overhead, etc. The cost of propellent is not shown above, as cost will normally be less than 0.1 cent per unit.

The above table illustrates that a difference of approximately 6 cents per unit exists between a 12 fluid ounce fill of barbecue sauce in glass and a pressurized spray can of the same product. It is felt by many that the pressurized can will cost between 6 to 10 cents per unit more than a companion product in glass or

* Presented at the 18th Annual Meeting of IFT, Chicago, May 27, 1958.

plastic. The decision remaining at this stage is whether the convenience and utility inherent in the pressurized package justifies the price difference when both products are displayed side by side on the grocer's shelf.

Preliminary testing and evaluation of the product by research personnel will do much to assist management and sales in the initial stages of evaluation. This preliminary testing would most probably be directed toward determination of desired dispensing and related product characteristics. The evaluation of dispensing characteristics could best be accomplished by one or both of the following methods:

1. Comparison of the existing product with samples of a like product in a pressurized package. In most cases, samples of like products will soon be available from a specialized development source; such as, the can companies, propellent manufacturers, valve manufacturers, or custom packager.
2. Submittal of an existing product to one of the above sources for a preliminary attempt at packaging.

Results of one or both of the above mentioned methods will indicate whether the existing product is applicable to the pressure package. The necessity of reformulation in terms of particle size, viscosity, or product stability should be readily apparent from this cursory evaluation. If it is felt by the combined forces of management, sales, and research that the product is a "natural" for pressurized packaging, the next step should logically be the institution of a developmental program.

Institution of the research program. The actual steps to be taken in the initiation of a developmental program for pressurized packaging appears to be of prime interest. The tremendous desire for a "first," as well as the push to compete with products that are currently appearing, should not curtail the need for proper product development. The age old phrase "haste makes waste" could not fit better than in today's race for new products.

Due to the newness and complexity of the pressurized food field, most food manufacturers are unequipped, both technically and mechanically, to conduct completely independent research projects. Thus, the present tendency is toward a combined project with one or more of the following specialized service organizations:

1. Major can companies
2. Propellent manufacturers
3. Valve manufacturers
4. Custom packagers
5. And other related organizations, such as the flavor and spice houses.

Each of the above mentioned service organizations has specialized experience in one or more facets of the pressurized food field. When a combined research project is initiated with one or more of these sources, there is a greater opportunity for ultimate success. The newness of the pressurized food field most probably precludes any one service organization from having a complete working knowledge of all aspects of the field. The qualified custom packager is probably in the best position, in most instances, to evaluate the various phases of pressure packaging. Certainly, the specialized knowledge that the food manufacturer has of his own product, when combined with the experience of the service organizations, offers far greater opportunities to bring the development project to a rapid and successful conclusion.

SEVEN BASIC FACTORS

Factors that must be considered are many and in most instances are interrelated. These factors may be roughly divided into 7 basic groups or phases, which are:

1. Product formulation
2. Propellent requirements versus product formulation
3. Valve requirements versus product formulation
4. Actuator requirements versus product formulation
5. Container requirements versus product formulation
6. Bacteriological requirements versus product formulation
7. Product end use

It would be virtually impossible to point out, in a paper of this length and scope, the many factors involved within each of the above phases. Thus, only the more significant factors in each of the 7 phases will be considered. At a later date, a "Pre-Marketing Check List for Pressurized Foods" will be available which will cover in detail the factors to be considered.

Product formulation. The importance of a product tailored to the requirements of a pressurized package is most important in achieving successful dispensing characteristics. It has been proved to be far more difficult to adapt a pressurized package to the product. Product characteristics that control the ability to successfully dispense a food as a spray, foam, or stream must be accurately tailored to the pressure package. Factors of particle size, viscosity, pH, color, and flavor are those considered as most critical in achieving the desired dispensing characteristics.

The factor of particle size control has become most significant in the field of spray foods. The limits within which particle size must be controlled are those set by the diameter of the smallest orifice in the valve system. It is most important to insure that the largest particle in the product is reduced to a size far less than that of the smallest orifice. The presence of large particles in a spray actuator will result in clogging or permanent distortion of the spray pattern. Use of oils of spices, liquid sugars, wines, and fine finishing techniques will do much to insure particle size control.

Control of product viscosity is of the utmost importance in achieving the desired dispensing characteristics *(7)*. Necessarily, a product to be spray applied must be thin enough in consisteney so that it can be broken up by the mechanical actuator. Prod-

ucts of high viscosity must normally be reformulated to the point where product flow within the container is at least equivalent to the rate of product removal. In many instances modification of viscosity by homogenization or additives will aid in attaining the desired dispensing characteristics.

Product color and flavor are quality attributes that must necessarily receive much consideration. Modification of a product in terms of particle size and viscosity will, in some instances, modify the basic color and flavor of the product. The culinary art of the food technologist is invaluable in compensating for these changes. The effect of the propellent employed can modify color characteristics. Thus, a product that is adversely affected colorwise by pressure packaging will employ a propellent, such as nitrogen, which will minimize deleterious changes in color.

Propellent requirements versus product formulation. The development of a product-propellent relationship for dispensing a pressurized food product depends primarily upon the desired dispensing characteristics. There are several propellents that are currently acceptable for packaging pressurized food products. They are nitrous oxide, nitrogen, argon, and carbon dioxide. In the relatively near future it is also felt that liquefied propellents, such as Freon C-318, may be approved for certain food uses, assuming it is competitive costwise *(3, 5)*.

The choice of one propellent versus another is governed by several factors, the primary ones being dispensing characteristics and solubility. If the product is to be dispensed as a foam or spray, a gas with high solubility, such as nitrous oxide, would be employed. If the product suffers deleterious color or flavor changes when exposed to high solubility gases, a low solubility propellent, such as nitrogen, should be employed. The type of dispensing characteristics desired will do much to dictate the degree and seriousness of color changes.

The relationship of factors, such as equilibrium, product fill, and temperature, should be determined and correlated for each product. Variations in fill influence the percentage by weight of propellent that can be introduced. This is of particular significance in products where foaming is to be achieved by the use of more soluble compressed propellents. The relationship of charging pressure, either with or without agitation, is of vital interest in achieving the desired dispensing characteristics, as well as assuring adequate removal of product.

Higher solubility in a product that is to be spray dispersed is in most instances desirable. The aeration of the product is usually not as obvious when the product is sprayed. The additional safety factor of increased gas weight in the container must be weighed against the possibility of slight color change in a spray food product. Increased gas weights within the container also offer a limited advantage in assisting break-up of the product.

The use of blended gases should also be considered in products where straight gassing does not give desired characteristics *(7)*. Blending of gases to balance flavor characteristics has long been used in the pressurized whipped cream industry *(3)*. It has now been shown that new product characteristics are available by blending two gases of differing solubilities.

Valve requirements versus product formulation. The development of a valve system for a pressurized food package should be based upon attainment of the following factors:

1. Desired dispensing characteristics
2. Freedom from clogging
3. Correct delivery rate
4. The correct dip tube length and positioning in the container.
5. Enamel coatings, and corrosion resistant fitments.

Achievement of desired dispensing characteristics will to a large degree rest with the choice of the proper valve system. If a spray application is desired, the valve choice is limited, at present, to the dip tube valve and mechanical actuator combination. Where a non-aerated stream is desired, either a "nozzle down" or dip tube type valve may be employed. Foam characteristics with soluble propellents may be achieved with either the "nozzle down valve" or the dip tube type valve. The choice as to "nozzle down" or inverted dispensing or upright dispensing rests with the food manufacturer. Each form of dispensing has its advantages and disadvantages, and can best be judged by the food manufacturer.

The valve system in a pressurized food, whether a spray, foam, or stream application, serves the basic purpose of guiding the product plus propellent to the actuator. The valve should be so constructed that there is a minimum of restriction to product flow. It has been shown that the use of large orifices in the valve system is of benefit not only from the standpoint of freedom from clogging, but in attaining desired dispensing characteristics. Modification of the valve orifice diameter will aid primarily in alteration of the flow rate and hence the delivery rate.

The valve cup must be so designed to withstand the attack of corrosive products. Development of specific enamels and methods of enamel application will be necessary to attain long shelf life with certain corrosive food products. The effect of temperature on the plastic valve components is also a necessary consideration.

Actuator requirements versus product formulation. The development of actuators for spray application has become one of the more important phases in the development of new pressurized food products. Due to the nature of the compressed propellents, the break-up of the product can be achieved only with the assistance of the mechanical break-up actuator. Such is not always the case in the non-food aerosol field. In many non-food aerosols the break-up is achieved not by the actuator but by the physical expansion of the propellent as it leaves the container. The end result is atomization of the product.

The actuator to be employed in spray-dispensing of foods must be specially constructed to achieve the desired spray pattern, particle size, and delivery rate.

Each product will usually require specific modifications of the already existing "mechanical break-up" actuators. Modifications of orifice size, spinner or swirl patterns, and expansion chambers may be so employed to achieve desired delivery rate, spray pattern width, and particle size. The combined efforts of the valve and actuator manufacturers will be required to determine the most advantageous actuator-product relationships.

A product to be dispensed as a non-aerated stream will employ either an upright valve and spout type actuator or a "nozzle down" valve. The spout type actuator serves basically as an extension for the guiding force the valve lends to the product and propellent. Non-clogging, drip-proof, and uninterrupted delivery is the goal of new type actuator development. Most of the spout type actuators available posses one or more of the above characteristics and are readily adaptable to stream dispensing of many varied products.

Container requirements versus product formulation. Determination of compatability between product and container is perhaps one of the most important phases in the development of a new product. This is especially true in the field of pressurized foods. Due to the newness of the field, there is a lack of data on container systems for packaging pressurized foods.

The product to be tested should be packaged in a range of container variables that are applicable to the product type. The packaged product should then be stored under controlled conditions of time and temperature. The test variables should be stored in both the upright and inverted positions at 70° F. and 100° F., and examined at regular intervals. During these periodic evaluations, which should ideally be carried on in conjunction with a can company, particular attention should be paid to the following factors:

1. Effect of container components on product quality in terms of color, flavor, pH, and stability loss.
2. Effect of product components in terms of enamel and/or tinplate corrosion or failure. The effect of product on failure of side striping materials, side seam, and double seam structures should also be evaluated.
3. Effect of product or vapor phase on the valve components and actuator in terms of corrosion and valve failure.

The evaluation of container-product relationships should also yield invaluable information on propellent seepage rates under controlled storage conditions. The effect of time and temperature upon dispensing characteristics such as spray patterns, delivery rate, and overrun will also be evident during these periodic examinations.

Bacteriological characteristics versus product formulation. Compilation of data on the bacteriological characteristics of a food product to be pressure packaged is essential in assuring lasting shelf-life and quality. The newness of this form of food packaging accentuates the need for complete and reliable data on the bacteriological stability of product during the various phases of packaging.

Factors of product composition such as moisture, ph, total solids, and inhibitors—such as salt or acid—aid in determining the method of preservation and conditions under which the product should be stored. It is essential in evaluating any new food product to determine the effect of representative florae in the product under various storage conditions. One method of accomplishing this is the use of inoculated packs. Inoculation of pure or mixed cultures of representative florae should indicate:

1. The method and degree of preservation required to assure stability.
2. Fill temperature.
3. Thermal death time characteristics.
4. The effect of inhibitors; such as salt, acids, or other chemical preservatives.

Controlled bacteriological storage studies and inoculated packs will also yield invaluable data on the relative merits of hot or cold charge methods. Weak points or points of recontamination may also be observed during the course of these studies.

Completed product use test. A complete evaluation of the finished packaged product should be conducted before the product is deemed ready for introduction to the consumer. Evaluation steps are best conducted by the combined forces of management, sales, and research. Impartial evaluations by outside sources of the product use and application will also aid in evaluating the relative merits of the new product. The ideal situation would be an objective evaluation free from the effects of over enthusiasm.

The evaluation of product application, convenience and quality should be based upon some of the following factors:

1. Ease of operation.
2. Effectiveness of product application.
3. Development of instructions and label (directions and cautions).
4. Product spillage, drip or splattering, and possible adverse effect on materials in the home.
5. Product color and flavor in the finished product.
6. Sales appeal of container and related exterior components.
7. The presence of a tamper proof seal, stacking and pricing feature.

Completed product evaluations should also be directed toward determining the acceptability of the container in terms of existing government regulations. The various agencies of the government; such as, the Food and Drug Administration, U. S. Department of Agriculture, and Interstate Commerce Commission, have specific regulations pertaining to the packaging, handling, storage, and shipment of pressurized products.

The significance of these evaluations will be ultimately shown when the product is in the consumers'

hands. Thus, the evaluation, packaging, and subsequent introduction to the retail market should be conducted in a careful and flawless manner.

Packaging the finished product. The decision to package for test marketing and, assuming consumer acceptance, for commercial distribution raises many questions. The most prominent ones being, "how and where is the product to be packaged?" The food manufacturer who does not possess equipment or facilities for packaging this new type of product is faced with one of two choices:

1. Install packaging facilities for pressurized foods.
2. Have product packaged by a qualified custom packager.

The choice as to one of the above alternatives will be governed by such factors as time, economics, product quality, and production. Unfortunately, there is no rule of thumb that can be employed in deciding whether or not to establish packaging facilities. The relative merits of each course of action must be weighed against the market potential of the product, the capital investment, and availability of experienced personnel before a decision can be made.

The qualified custom packager offers many advantages to the food manufacturer. The versatile production, research, and quality control facilities are more readily available at short notice to the food manufacturer. The qualified custom packager can be advantageously employed in initial packaging stages while the acceptance of the product is being determined.

Each and every food manufacturer who considers entering this field must bear in mind that he is ultimately responsible for the quality of his product. The qualified custom packager offers much background and experience developed in the pressurized non-food field. This knowledge, when merged with that of the food manufacturer and other service organizations, offers the shortest and safest route to a successful package. If at any time the food manufacturer finds that the factors of capital investment, time, experience, and economics are favorable, he may select to install his own packaging facilities.

SUMMARY

Need for a properly guided program from inception of the product to final consumer usage is vital to all industries, and especially to the new pressurized food field. The various phases of pressurized food packaging and development are allied; yet ,in many cases, foreign to the average food manufacturer. Thus, the food manufacturer is dependent upon the knowledge and experience available from the various service organizations. A merging of knowledge from the food, valve, propellent, and container manufacturer, as well as the custom packager, is vital in assuring the quality and success of the pressurized food field.

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The Food Processor in the Pressurized Food Field*

(Manuscript received May 11, 1958)

Victor J. DiRocco
Heublein, Inc., Menlo Park, California

CONTINUING RESEARCH has now made it possible to dispense food products in a variety of forms and, in so doing, promises to add still another successful chapter to an already soaring specialty business. The food processor is constantly striving to produce new products. When this cannot be achieved, he must be content to settle for a new "improved" presentation of an already proven or established product. It should be recognized, however, that relatively few products introduced are actually innovations; they are, in reality, products given the "new look" treatment by new packaging concepts. Pressurized food packaging, for example, now makes it possible to introduce many products in a new dress, thereby enhancing their acceptability.

The pressurized food field presents a tremendous potential to the specialty food manufacturer. Pressurized foods afford the specialty food processor a full complement of natural attributes for successful merchandizing; namely, (1) extra high quality that will justify the higher price tag, (2) convenience and (3) attractive packaging, with sales appeal.

CASE HISTORY OF A PRESSURIZED BARBECUE SAUCE PRODUCT

Considerable thought was given to our pressurized barbecue sauce product. It appeared to possess a convenience feature and utility, but we wished to avoid its being labelled "a gimmick." We are not interested in manufacturing "gimmicks" for we must consider not only the immediate, but also the long-term appeal. Our product is somewhat different from the ordinary sauces in that it is a sweet-sour type sauce. It appears to be a "natural" in that it offers distinctive character in flavor and usage.

Preliminary evaluations of more than 40 barbecue sauces from different areas of the country indicated:

1. There was no one sauce that possessed flavor characteristics which would make it acceptable in all areas of the country.
2. The door was open to the introduction of a sauce that would possess distinctive flavor and sales appeal to the barbecue conscious consumer.

The sweet-sour sauce decided upon is of a Polynesian type, long associated with the true or original art of barbecuing. The subtle blending of Pineapple, A-1 Sauce, sugar and other ingredients brings the American barbecuer, as well as the gourmet, a new sensation in flavor.

Working with the research facilities of a major can company, preliminary investigations were successfully made utilizing compressed gasses as the propellent. It was recognized at this point that many problems would have to be resolved before this product was to become a reality. Ideally, it would be simpler to formulate to suit the available packaging components. However, the problem was somewhat complicated by the fact that the barbecue sauce formulation had been established previously, thereby necessitating a complete evaluation of all relationships; i.e., can structure, pH, viscosity, particle size, propellent, valve, actuator, delivery rate and dispensing characteristics.

Once the product and propellent relationship had been established, a suitable valve and spray head, or actuator, had to be designed since it was desired to have the product dispensed as a spray rather than as a stream. In order to accomplish this, modifications in the product formulation had to be made in order to control product particle size and viscosity. Improved methods for product finishing had to be devised in order to assure clog-free passage of product through a valve-actuator system with a limiting orifice diameter of .016 thousandths of an inch. This had to be accomplished without sacrificing product flavor, quality, or appearance.

As an acid product with a pH of approximately 3.5, a great deal of attention had to be given to the composition of can linings and valve cups and their ability to withstand corrosion. In conjunction with this study much effort and consideration were given to the residual oxygen content in an effort to minimize its effect on the product, as well as the can lining system. Various can systems were evaluated under controlled conditions of time and temperature. This investigation also included:

1. The use of antioxidants; such as, glucose-oxidase catalase enzyme systems.
2. The purging of cans with nitrogen or carbon dioxide prior to and after filling.
3. Various levels of vacuum, prior to crimping.

Stability. As a result of shelf-life studies, it was found that maximum shelf-life could be obtained by filling at 190° F. and mechanically drawing a vacuum in excess of 20 inches prior to the actual crimping operation. It was further determined that by maintaining a residual oxygen content of less than 1%, a satisfactory shelf-life was obtained with a plain 1.25 H.D.T. container.

Inoculated test packs were conducted to assure adequate processing procedures. As an acid product with a pH less than 4.5, it was felt that a filling temperature of 190° F. after pasteurization at 200° F. would be adequate to prevent spoilage by yeasts, molds and acid-forming organisms, all of which are known to have a relatively low heat resistance. The product was inoculated with a mixed flora at various

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points of possible contamination in the operation. Particular attention was given to the gassing operation. Assuming that the filled cans are sterile, it is the last step in the operation that is susceptible to possible recontamination. To date tests have proved to be negative. The findings are to be reported at a later date.

Finding a packer. Our next step was to find a custom packer who might be willing to work with the processor in tackling the many yet unsolved related problems before commercial production of the product was to become a reality. The problems included design and installation of equipment to prepare the product for this specialized form of packaging.

Although it took a year's research, the necessity and importance of thoroughly investigating the many facets of a program is understandable. It was recognized from the outset that the final product would in effect represent the possible summation of solutions to many technical problems. Having achieved this, equal emphasis was given to setting up a thorough and far reaching quality control program, particularly since the product was to be packed by a custom filler.

INTRODUCING THE PRODUCT

Introduction of this product to the trade had to be accomplished in as flawless a manner as possible. The product was introduced in several large southwestern cities for the most critical test any new product must undergo—consumer acceptance. Test market results were quite favorable. Results from the market tests tend to verify, in fact, the optimistic views that many have for the tremendous potential of this new field. Experts in this field now predict production figures of 500,000,000 units per year by the year 1959.

The recent introduction of this product, along with a chocolate syrup, has understandably aroused the interests of many food processors. Those contemplating production of a pressurized food product will wisely seek the specialized technical assistance of the major can companies, propellent and valve manufacturers as well as the custom packers. To date there are only a few custom fillers in a position to pack pressurized foods; however, it is merely a question of time before the many non-food custom packers will be installing food lines. In such cases, it will become the food technologist's responsibility to make good food processors out of the custom fillers almost overnight—this in order to protect the consumer, the packer, and the industry. Competition in this field will undoubtedly become keen and many will attempt to shortcut operational costs which will no doubt manifest themselves in an inferior product. The individual food processor, as well as the food industry in general, cannot and must not tolerate any compromise with product quality.

Although there are many sound arguments for having a pressurized food product custom filled—and the custom filler is certainly capable of an excellent job—the food manufacturer, regardless of the size of his operation, should consider doing his own packaging. By so doing he will not only have a better control of product, but also a more workable knowledge of purchasing and production costs.

SUMMARY

As a result of this study, the first spray type pressurized food product has been successfully marketed, thereby introducing a new trend in convenience type packaging. The importance of thoroughly investigating the economics, as well as the technical aspects, before commercially producing a pressurized food product cannot be overemphasized. As to whether or not the consumer will be willing to pay a higher price for the pressurized product, it is considered that the combination of excellent product quality, convenience, and sound merchandising will be the answer to this problem. It is felt that once the consumer has become sufficiently acquainted with the convenient specialty type products, introduction of staple items will become less difficult.

Acknowledgment

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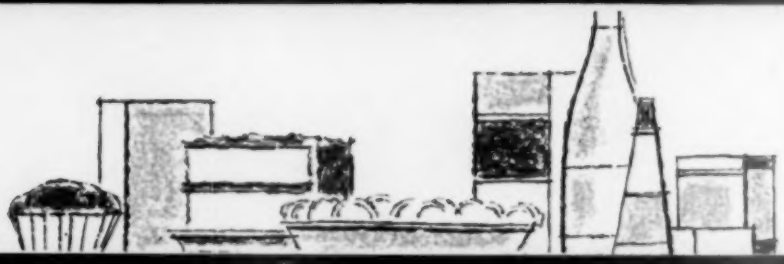

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Enterococcus-Like Organisms in Citrus Juices*

Roger Patrick and E. C. Hill

Florida Citrus Experiment Station,
Lake Alfred, Florida

(Manuscript received February 8, 1958)

REPORTS OF THE OCCURRENCE of streptococcus-like organisms in citrus juice stimulate interest in the mode of entry of these organisms into citrus juices and their possible significance with respect to public health. The purpose of this study was to isolate such organisms, to obtain further insight into their mode of entry and the sources of contamination, and to explore their effect on health.

Kaplan and Appleman *(8)*, the first to report the occurrence of such organisms in citrus products, isolated 73 strains of enterococci from frozen concentrated orange juice. These strains survived 60° C. for 30 minutes, were able to grow at pH 9.6, and also grew in the presence of 0.1% methylene blue. They grew at 45.5° C. and in the presence of 6.5% sodium chloride. Gelatin was liquefied by 63 of the cultures; these they designated as *Streptococcus liquefaciens*. The remaining 13 cultures were classified as *S. faecalis*. The previous history of the samples from which these isolations were made was not given. Frequently, it is not possible to obtain this information, but its usefulness in interpreting the data needs no elaboration. Farraro and Appleman *(5)* examined 9 brands of orange concentrate and found all samples positive for enterococci. The previous history of these samples was also not given. The authors suggested the need of continuing the study to explain the uniform presence of enterococci in frozen orange concentrates.

Allen and Fabian *(1)* reported that *S. faecalis* remained viable longer than *Escherichia coli* in acid foods such as orange juice, pH 3.5, held at 30° C. for 7 days. They did not consider their presence in moderate numbers as a sure sign of dangerous contamination, but rather of possible contamination. They suggested that a search for the sources should be made. Other investigators have observed the hardiness of enterococci in acid foods. Hahn and Appleman *(6)* reported that a strain of *S. faecalis* survived 72 hours at −17° C. in orange concentrate, but enteric rods could not be recovered after 24 hours. Because of the complexity of the problem, they concluded that *S. faecalis* should not be used as an indicator of pollution. Hahn and Appleman *(7)* also found that *S. faecalis* was destroyed when the peel oil content of orange concentrate was increased to 1,000 p.p.m. They observed that when concentrate was stored at 4° C. the bactericidal action of citric acid was greater than hydrochloric acid when added at comparable hydrogen ion concentrations. The acid exerted a more toxic effect than was indicated by the degree of dissociation. Larkin, Litsky, and Fuller *(10)* inoculated orange concentrate with *S. liquefaciens* and *S. faecalis*, stored it for 147 days at −10° F. and found that the numbers remained viable and constant. This concentrate had also been fortified with 4% citric acid, pH 3.0. They concluded from these observations that "Fecal streptococci could be used advantageously in preference to coliform bacteria as indicator bacteria in frozen food."

Concerning the significance of enteric organisms in citrus juices, Larkin, Litsky, and Fuller *(9)* found fewer enterococci in citrus concentrates than coliforms in frozen vegetables, but the authors concluded that since frozen vegetables are cooked, coliforms were of little importance, but that even a few enterococci in citrus concentrates should presuppose great significance as a health hazard. They emphasized the fact that fecal bacteria in any food constitutes a possible health menace and that a wide-spread epidemic could result from frozen foods that have not been processed properly.

Dack *(3)* reminds us that millions of cans of frozen concentrated orange juice have been consumed without causing any known cases of enteric infections. Vaughn, Murdock, and Brokaw *(13)*, reviewing the significance of fecal streptococci as indicators of pollution, concluded that because of their universal presence, they probably are not good indicators of sanitation. They found no reported evidence on fecal streptococci indicating that it would grow in frozen citrus products. Sherman *(12)* pointed out that *S. faecalis* and *S. liquefaciens*- types of enterococci occur commonly on surfaces of plants. This may mean that these organisms are merely surviving rather than growing under these conditions and are readily distributed from the fruit surface to the juice. Also, it has been noted that none of the hemolytic-type of enterococci have been isolated from plant materials. Sherman also noted that some of the species of enterococci are separated from each other by rather thin and shaky boundaries. There is no concrete evidence of a health hazard due to the presence of streptococci and possibly enterococci in citrus juices or frozen citrus concentrates.

At the Citrus Experiment Station for the past 4 years, we have been associating streptococcus-like contamination with the quality of the fruit. We were not interested primarily in the identification of all streptococcus-like organisms in the citrus juices, only those which were possibly of an enteric nature. The group is well defined as to their reactions under stated conditions, Bergey *(2)*. They will grow at 45° C. in 6.5% sodium chloride, tolerate 0.1% methylene blue, survive 62° C. for 30 minutes and will grow at pH 9.6. We applied the same measures to the streptococcus-like bacteria found in citrus juices and selected for a

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more detailed study those organisms which gave a confirmed test for the enterococcus group.

EXPERIMENTAL

During a survey conducted in the 1956–57 season, examinations were made of 212 samples of frozen concentrated orange juice from 24 processing-plants and 99 frozen concentrated grapefruit juices from 15 processing-plants. Samples of frozen concentrated juice were obtained semi-monthly throughout the processing season. Streptococcus-like organisms were found in 191 of the orange and in 29 of the grapefruit samples, a total of 220 out of 311 samples investigated. The previous history of these commercial samples is not known too well. It is known that some fruit which had been damaged previously by cold weather was processed. In addition to the commercial samples, 34 samples of orange juice were examined from lots of cold-damaged oranges freshly extracted in the Citrus Experiment Station's pilot plant; the history of these specimens is well established. Twenty-four of these samples contained streptococcus-like organisms, thus making a total of 345 samples examined, with 244 (71%) of them containing streptococcus-like bacteria.

These organisms were isolated after inoculating 20 ml. of lauryl tryptose broth with 10 ml. of a 1–10 dilution of reconstituted juice and incubating the tubes at 35–37° C. for 48 hours. If streptococcus-like organisms grew, transplants were made to Bacto-enterococcus confirmatory agar, Difco *(4)*. This medium served for screening purposes, and the cultures that grew were subjected to a more complete confirmatory test. A broth medium was made of the above formula, plus 65 g. of sodium chloride per liter and, just before using, 65 units of penicillin were added to each 100 ml. of broth. Enough of this broth was added to each newly inoculated slant to cover approximately one-half of the surface. Incubations were made at 45° C. If coccus-shaped Gram-positive organisms grew in this medium at 45° C. and were catalase negative, they were generally considered confirmed positive evidence of enterococci. Of the 244 streptococcus-like cultures, 26 (11%) grew in the screening medium and 13 of these, 5.5% of the total number examined, survived the more complete confirmatory test. These 13 cultures were subjected to other biochemical tests to identify them more completely.

RESULTS AND DISCUSSION

Thirteen streptococcus-like cultures grew at 45° C. in the presence of 6.5% sodium chloride, were catalase negative, and were considered Gram-positive. The status of the latter test was doubtful, however, for some strains. These strains also produced ammonia in 4% peptone water adjusted to pH 7.0 when incubated at 45° C. Growth and ammonia were produced in the same medium adjusted to pH 9.6 and incubated at 45° C. They grew in milk containing 0.1% methylene blue and reduced the dye when incubated at 37° C. or 45° C. They also grew in lactose broth with 10% bile and gave a methyl-red reaction (pH 4.0 to 4.5) in lactose and glucose broths. They did not hydrolyze starch, inulin, salicin, hippuric acid or esculin. Mannitol and trehalose were fermented, but raffinose was not fermented. All of these reactions are characteristic for the enterococcus group.

Some irregularities were observed. The 13 cultures grew more readily at 45° C. than at 37° C., and none grew profusely on the surfaces of solid media. When the 13 cultures were heated at 62° C. for 30 minutes, 7 of them failed to survive; enterococci are expected to survive this test. However, other characteristics of these 7 cultures make them comparable to *S. liquefaciens*. They grew and produced ammonia in 4% peptone water at pH 9.6 at 37° C., liquefied gelatin, and fermented sorbitol. Tubes of litmus milk inoculated with these organisms reacted in uniform agreement: the dye was reduced, peptonization began within 24 hours and continued to digest the weakly coagulated solids almost completely. The proteolyzed milk was not bitter to taste as is characteristic of *S. liquefaciens*. Milk with 0.1% methylene blue did not show coagulation, indicating that methylene blue may not be substituted for litmus in milk culturing. In spite of some irregularities, shown in Table 1, the characteristics observed show a great similarity to those of *S. liquefaciens*.

The 6 cultures which survived 62° C. for 30 minutes failed to grow in 4% peptone water at pH 9.6 when incubated at 37° C., which may be an irregularity of some significance. When inoculated into litmus milk, they readily reduced the indicator which oxidized slowly later, produced acid, and formed a firm coagulum. Coagulation was not observed when the cultures were inoculated into milk containing 0.1% methylene blue. Table 1 shows the similarity of the characteristics observed to those known for *S. faecalis*.

The source of this kind of contamination seems to be the raw fruit from the grove. Contamination is encountered regardless of whether the fruit is selected with care or obtained by grove run harvesting. The degree of contamination is dependent upon its previous history. Cultures 3–24, 25, 26, and 27 were isolated from fruit that had been damaged by low temperatures and left on the trees for about 4 months. The juice was extracted in the Citrus Experiment Station's pilot plant where sanitation was exceptionally good. The remaining 9 cultures came from commercial survey samples. Isolations were made from these samples about 3, 4, and 7 months after the first possible time of freeze damage.

Although the fruit receives a thorough washing, the juice extracted from freeze-damaged, over-mature, or mishandled fruit often contains contamination of a streptococcus-like nature. Juice extracted from undamaged fruit of prime or under prime maturity has not been observed to contain streptococcus-like contamination. Single strength juice containing these organisms passing through a plate-type heat exchanger contaminated the cooling side, so that the processed juice possessed a dense population of streptococcus-like organisms. How this contamination is effected is a matter of speculation. The condition was verified by swab tests in which the swabs were immersed and incubated in lauryl tryptose broth. It is necessary at the Citrus Experiment Station to check the cooling side of the plate-type heat exchanger for this kind of contamination after each clean-up period; however, these streptococcus-like organisms are easily killed by heat. This fact was observed after the heat exchanger had been cleaned thoroughly and checked for microbial contamination. Juice from freeze-damaged Pineapple oranges possessing streptococci were heat treated in a plate-type heat exchanger to 205°, 195°, 185°, 175°, and 165° F. adjusted to a retention time of 14 seconds; no streptococcus-like con-

TABLE 1

Some cultural characteristics of the streptococcus-like organisms that survived the enterococcus-confirmatory test

Culture numbers	Gram's stain	Ammonia from 4% peptone water		Δ 62° C. 30 min.	Gelatin	Sorbitol	0.1% Methylene blue milk		Ulrich milk	Litmus milk	Glycerine	Sucrose
		pH 7.0 at 45° C.	pH 9.6 at 37° C.				at 37° C.	at 45° C.	at 37° C.	at 37° C.		
2–33	—	+	+	—	+	+	RP	RP	RP	RPC	+	+
2–39	+	+	+	—	+	+	RP	RP	RP	RPC	+	+
2–56	+	+	+	—	+	+	RP	RP	RP	RPC	+	+
3–7	+	+	+	—	+	+	RP	RP	RP	RPC	+	+
6–34	—	+	+	—	+	+	RP	RP	RP	RPC	+	—
6–35	+	+	+	—	+	+	RP	RP	RP	RPC	—	+
6–43	+	+	+	—	+	+	RP	RP	RP	RPC	+	—
S. liquefaciens	+	+		+	+	+	R			RACP	+	+
							RO	RO	RO	RAC		
2–69	—	+	—[2]	+	—	—	RO	RO	RO	RAC	+	+
3–24[1]	+	+	—[2]	+	—	—	RO	RO	RO	RAC	+	+
3–25[1]	+	+	—[2]	+	—	—	RO	RO	RO	RAC	+	+
3–26[1]	+	+	—[2]	+	—	—	RO	RO	RO	RAC	+	+
3–27[1]	—	+	—[2]	+	—	—	RO	RO	RO	RAC	—	+
3–66	—	+	—[2]	+	—	—					—	+
						—					—	—
S. faecalis	+	+		+	—	+	R			RAC	+	+

[1] Cultures isolated from raw juice extracted from frozen fruit at this Station.

[2] Failed to grow.

Key: RP = Reduced, proteolyzed; RPC = Reduced, proteolyzed, coagulated; RAC = Reduced acidified, coagulated; RO = Reduced, oxidized; RACP = Reduced, acidified, coagulated, proteolyzed.

tamination grew in inoculated lauryl tryptose broth after the heat exposure to 165° F. Murdock, Brokaw, and Allen *(11)* also found that this type of heat exchanger was a source of contamination in processing juices for frozen concentrate. They found a strain of *Lactobacillus* that accumulated in the cooling side of the exchanger and was capable of producing an off flavor in the juice. It has also been shown through tests at this Station that the streptococcus-like contamination did not survive the process of concentration. Many of the commercial concentrates gathered in this area during the past season might have been diluted to 42° Brix with heated juice that had been contaminated with streptococcus-like organisms from the cooling plates of the heat exchanger. Evidence of this is that there were no viable *Escherichia* or *Aerobacter* bacteria found and streptococcus-like organisms predominated when inoculations were made into lauryl tryptose broth.

Since these data were collected in the citrus producing area where the facts were gathered first hand, it seems highly improbable that the streptococcus-like bacteria observed came originally from filth in the processing plants. More likely they were surviving from the grove where the fruit was grown and thus reflected the quality of the fruit used.

SUMMARY

About 71% (244) of the samples of citrus juices analyzed for bacterial contamination contained streptococcus-like microorganisms; 11% (26) of the cultures survived the presumptive test for enterococci and about 5.5% *(13)* of the cultures survived the confirmatory test. Seven of the cultures were believed similar to *S. liquefaciens*, even though they failed to survive the heat test of 62° C. for 30 minutes, failed to coagulate milk in the presence of 0.1 per cent methylene blue but did reduce the indicator, did not produce acid in litmus milk, and did not impart a bitter taste to the proteolyzed casein. The remaining 6 cultures failed to grow at pH 9.6 at 37° C. and also failed to coagulate milk in the presence of 0.1% methylene blue. These are two of the irregularities observed when comparing the characteristics of these cultures with *S. faecalis*. Four of the 6 cultures were isolated from juice of cold-damaged fruit that was extracted in the Citrus Experiment Station's pilot plant where sanitation is exceptionally good.

Streptococcus-like organisms were eliminated from juice readily by heating for 14 seconds to 165° F. Commercial concentrates could have been reinfected with single strength juice contaminated from the cooling plates of the heat exchanger while in the preparation of a 42° Brix product from juice of a higher concentration.

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Biochemical Methods for Determining Shrimp Quality[a, b]

I. Study of Analytical Methods[c]

(Manuscript received December 21, 1957)

Marcel Gagnon[d] and Carl R. Fellers[e]

Department of Food Technology, University of Massachusetts, Amherst, Massachusetts

Biochemical activities of microorganisms in marine products have been extensively studied for a number of years. Measurement of the metabolic products is believed to be the answer to the search for estimating the degree of freshness of fish and meat products. Spoiling fish, being a proteinaceous substratum, yields products in varying amounts depending on numerous factors of microbiological, chemical, or physical origin.

It was the purpose of this study to investigate the decomposition products in shrimp, emphasizing mainly the determination of quality in frozen breaded shrimp. A comprehensive review of the literature was first undertaken on the tests and procedures pertaining to the subject. Laboratory work was carried out to determine their validity in detecting changes in composition of the protein fractions, together with corroborative organoleptic and bacteriological studies. Data obtained were analyzed and the most promising method was selected to be studied more thoroughly. Both bacteriological and organoleptic tests served as a basis for statistical evaluation.

The overall aim was to obtain a definite relationship between a chemical objective test and the degree of freshness of shrimp products. Also, the test sought will be of interest only if simple and rapid; *sine qua non* requirements for its use in inspection and production control.

The chemical tests for quality of meat and fish have been the subject of intensive research since the beginning of the present century. Among the chemical tests which have received the greatest attention from workers have been the ones used to determine trimethylamine in particular and volatile bases in general. Chemical tests for indole and skatole, tyrosine, histamine, hydrogen sulfide and miscellaneous other decomposition products have been tried as freshness indices.

On the basis of preliminary laboratory and practical work done prior to this study, many possible indices were rejected. Trimethylamine determined by the method of Dyer *(5)* was found to be of very limited value. It was useful in showing when the frozen breaded shrimp were completely spoiled, but it was not reliable in estimating the actual degree of spoilage before reaching this extreme stage. Indole tests by the distillation method described by Duggan and Strasburger *(4)* and A.O.A.C. *(1)* and the ex-

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[c] Contribution No. 1127, Massachusetts Agricultural Experiment Station.

[d] Present address: Alphonse Raymond Ltée, Montreal, P. Q., Canada.

[e] Present address: University Laboratories, Amherst, Massachusetts.

traction method described by Chernoff *(2)* proved equally unreliable. Iodine titration by the Truttwin method *(11, 12)* was found of no value when applied to frozen breaded shrimp spoilage. Determinations of ammonia by the aeration method of Folin *(6)* and the method proposed by Ota and Oshiro *(8)* proved to be valueless due to the great variations found between samples of a same lot and within replicates of the sample. In almost all these tests, it was found that the quantity of the decomposition product measured was so small, compared to the whole, that a slight variation in the water, protein or fat content could throw the final results from one quality class to another regardless of the actual quality of the product. The only test which showed promise was devised by combining the actual total nitrogen content values of a sample with a value representing the volatile bases as a whole. This test showed justification for further study and was correlated with total bacteria count and taste panel score for each sample studied. Thus actual quality of each sample was clearly established.

METHODS

Samples. Frozen breaded shrimp were prepared in a commercial plant, in accord with usual procedures and quality standards. The cases of 10 oz. packages of frozen breaded fantail shrimp were shipped in large insulated containers with dry ice to the University of Massachusetts, Amherst. All were solidly frozen upon arrival. Samples were stored at −21.7° C. (−7° F.) and kept undisturbed until the storage and laboratory tests were undertaken (usually less than 15 days).

On the basis of a statistical analyses of the results obtained during preliminary studies, 6 replicates were found to be required to obtain reasonably significant results with the tests used. It was decided to carry out the storage tests at a temperature of 3° C. (38° F.) because a storage temperature of 25° C. (77° F.) induced a much too rapid spoilage rate. Every 24 hours, seven 10-oz. boxes of shrimp were selected at random from the lot. One box was quick frozen for later taste panel tests and the other six were used to carry out the chemical and bacteriological analyses.

TABLE 1

Ammonia (NH_3) recovered from a standard solution of ammonium sulfate using the procedure and apparatus for determining volatile bases (VB)

Distillation No.	NH_3 present	NH_3 recovered	Recovery	Difference
	mg.	*mg.*	%	%
1	3.00	2.86	95.3	−4.7
2	4.00	3.68	92.0	−8.0
3	5.00	4.75	95.0	−5.0
4	6.00	5.60	93.3	−6.7
5	7.00	6.55	93.6	−6.4
6	8.00	7.57	94.6	−5.4
7	9.00	8.70	96.7	−3.3
8	10.00	9.70	97.0	−3.0
9	11.00	10.75	97.7	−2.3
10	12.00	11.70	97.5	−2.5

Each box was opened and samples were removed aseptically for bacteriological examination. The remaining contents of the box were passed twice through a food chopper. Duplicate chemical analyses were made on this finely chopped sample.

Total bacteria count. The frozen, breaded shrimp sample was weighed aseptically into a sterile glass mechanical blender jar, which contained 450 ml. of sterile water. The contents of the jar were then thoroughly comminuted for 2–3 minutes. The blended sample was allowed to stand for 2–3 minutes until the foam subsided. One ml. of the mixture was pipeted into 99 ml. of sterile water. The bottle was shaken about 25 times and further dilutions of 1: 10,000 and 1: 100,000 were prepared. Difco Bacto-nutrient agar (1.5%) was used for plating as recommended by Green *(7)*. All plates were made in duplicate and were incubated at 25 (±2)° C., [77 (±3)° F.] for 3 days. The plates containing between 30 and 300 colonies were counted and recorded. Results were expressed in "number of bacteria per gram of frozen breaded shrimp."

Moisture. The moisture content was determined by drying samples in an air oven at 100° C. (212° F.) for 24 hours.

Percentage of breading. Frozen breaded shrimp were selected at random and weighed. Shrimp were washed in running cold water until all breading material had been removed. Washed shrimp were blotted on paper towels and weighed. Percentage of breading was calculated from the weight of the breaded sample and the weight of the washed and drained product.

Total nitrogen (TN). About 1 g. of the chopped shrimp was put in a previously weighed short glass pyrex tube. Weight of the sample was obtained by difference. Use of a pyrex tube facilitated weighing and transfer to the Kjeldahl flasks without adherence of the sample to the neck of the flasks. The digestion mixture was prepared as recommended by Peters and Van Slyke *(9)* but the addition of copper sulfate was omitted. It was replaced by selenized granules added directly to the digestion flask. A few drops of Dow Corning Antifoam A Emulsion [1] effectively prevented foaming. The digestion was carried out for one hour or until the mixture was clear. After cooling and diluting to 100 ml. with distilled water, aliquots of 10 ml. were used for distillation in an all-glass micro-kjeldahl apparatus. After addition of 10 ml. of a 40 per cent NaOH solution, the mixture was distilled. The ammonia was collected for 3 minutes in a 4 per cent boric acid solution to which had been added a few drops of Tashiro's indicator (0.246 g. methylene blue and 0.375 g. methyl red and 300 ml. 95 per cent ethyl alcohol). It was titrated with 0.05N HCl solution. Results were calculated as grams of nitrogen per 100 g. of frozen breaded shrimp.

Volatile bases. Ammonia and other volatile bases were determined as Volatile Bases (VB) by the following method, which was both simple and rapid and proved to be the most acceptable.

The sample of frozen breaded shrimp was passed twice through a fine food chopper. Five-gram portions were weighed in waxed carton cups. Waxed carton cups were used to facilitate the washing of the sample from the container. Upon defrosting, frozen breaded shrimp became very sticky due to the presence of starch in the breading. The sample was washed into a Waring blender with 100 ml. of CO_2-free distilled water. The mixture was blended for 5 minutes at high speed and transferred quantitatively to a 500 ml. Pyrex tapered glass-stoppered distillation flask by washing with approximately 100 ml. of distilled water. The distillation flask was quickly connected to a condenser after the addition of approximately 10 ml. of a 40% NaOH solution, boiling stones and a few drops of Dow Corning Antifoam AF Emulsion. The tip of the condenser was dipped in approximately 15 ml. of 4% boric acid solution in which Tashiro's indicator had been added. The distillation was carried out with a low flame and lasted 5 minutes after the appearance of a green color. The tip of the condenser was rinsed with CO_2-free distilled water and the distillate was titrated with a 0.05N standard HCl solution. The results were calculated as milligrams or grams of NH_3 per 100 g. of frozen breaded shrimp. Results from recovery tests carried out with this method are shown in Table 1. The average recovery was found to be 95.0%.

Ratio of volatile base (VB) to total nitrogen (TN). Results obtained for volatile bases (VB) and total nitrogen were used to calculate a ratio. In order to facilitate the calculations the ratio was multiplied by 100.

Organoleptic tests. A laboratory preference method was used. A hedonic scale was worked out which varied between 1—*like extremely* and 10—*decomposed*. The numerical values were omitted from the scoring sheet and only descriptive expressions

[1] Dow Corning Corporation, Midland, Michigan.

were used. The scores for each sample were given in terms of average score points awarded by the judges. The panel of judges was composed of trained staff members and graduate students of the Department of Food Technology.

The shrimp were removed from the packages and allowed to thaw until they could be easily separated. They were then placed in a wire frying basket, dipped into preheated vegetable oil at 185° C. (365° F.) and cooked for 2 to 3 minutes. The fried breaded shrimp were allowed to drain and were judged while still hot.

RESULTS AND DISCUSSION

Results for the whole study made on a lot of frozen breaded shrimp stored at 3° C. (38° F.) are given in Table 2. The average of 6 replicates at each storage time was calculated after eliminating extraneous values by means of the Q test *(3)*. Statistical methods *(10)* were used to determine whether or not a correlation between the factors studied does really exist. Thus, it became possible to ascertain the importance of each factor on the over-all finished product.

TABLE 2

Effect of storage at 3° C. (38° F.) on the chemical bacteriological and organoleptic determinations on a lot of frozen breaded shrimp[1, 2]

Storage time in hours	Average taste panel score[3]	Log of total bacteria per gram	Moisture	Total nitrogen	Volatile base (VB) calcd. as NH_3	Ratio of VB to total N x 100
			%	*g./100 g.*	*g./100 g.*	
0	2.2	5.91	63.2	1.88	0.116	6.2
24	3.0	5.36	64.5	1.77	0.122	6.9
48	3.3	5.72	63.4	1.76	0.131	7.4
72	3.9	5.58	63.9	1.73	0.128	7.4
96	4.2	6.10	60.9	1.77	0.129	7.3
120	5.0	5.92	62.4	1.75	0.126	7.2
144	6.2	8.30	62.7	1.77	0.150	8.5
168	5.2	7.63	63.5	1.76	0.135	7.7
192	5.5	8.46	62.0	1.76	0.154	8.8
226	6.3	8.71	62.4	1.75	0.172	9.8
240	10.0	9.35	63.8	1.58	0.158	10.0

[1] All the results given in this table, except for the average taste panel score, were obtained by averaging the results from duplicate analyses of six samples at each storage time.

[2] The average amount of breading for the lot was found to be 47.0 per cent.

[3] For the average taste panel score: L.S.D., 5% = 1.09; L.S.D., 1% = 1.38.

The numerical values of the hedonic scales for the organoleptic tests were averaged for each sample and are given in Table 2. By means of Tukey's method *(13)* of analysis of variance by ranges, the least significant difference (LSD) was calculated for the 1 and 5% level. A taste panel score of 5.0 to 6.0 indicated a questionable quality of the sample. In this experiment, the average taste panel score was used as a basis for comparison with the different tests carried out as shown in Figure 1.

The average taste panel score of 2.2 was found for the control sample indicating a very high quality shrimp product. A constant increase in the score was obtained until a value of 5.0 was reached. It fluctuated between 5.0 and 6.0 and increased rapidly to reach a score of 10.0 for inedible shrimp. Table 3 summarizes the analyses of the correlation shown by the data.

The total bacteria count had a lag period for the first 5 storage days, followed by a sharp increase which continued until total decomposition had occurred.

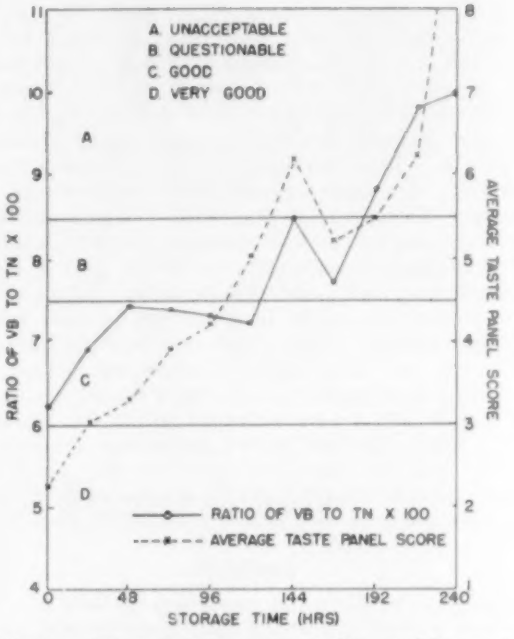

Figure 1. Effect of storage time at 3° C. (38° F.) on the ratio of volatile bases (VB) to total nitrogen (TN) (x 100) and the organoleptic determinations carried out on a lot of frozen breaded shrimp.

The total bacteria count lagged in the vicinity of 1,000,000 up to the 5th day and increased during the intermediatry stages of spoilage at a rapid rate. This behavior rendered the test unsatisfactory for estimating the degree of freshness in frozen breaded shrimp. The only indication it gave in the homogeneous lot was to confirm other tests at the stages of very good and decomposed breaded shrimp only when the initial bacteria count was known. If not related to a known initial count even an actual extremely high count is of no meaning insofar as quality is concerned, it could reflect only on the degree of cleanliness with which the product was prepared. A high, positive correlation factor was found between total bacteria count versus storage time, average taste panel score and

TABLE 3

Correlation (r) found from the statistical analyses of the results of storage at 3° C. (38° F.) on the chemical, bacteriological and organoleptic determination on a lot of frozen breaded shrimp

Correlation of	r[1]
Storage time vs. average taste panel score	+0.901
Storage time vs. volatile base	+0.885
Storage time vs. ratio of volatile base to total N	+0.945
Storage time vs. log of total bacteria per gram	+0.913
Volatile base vs. average taste panel score	+0.779
Ratio of volatile base to total N vs. average taste panel score	+0.894
Log of total bacteria per gram vs. average taste panel score	+0.861
Log of total bacteria per gram vs. volatile base	+0.908

[1] For a degree of freedom, the following correlation coefficients are given: 5% level: 0.602; 1% level: 0.735.

volatile bases, but in no way did the high degree of correlation change the usefulness of the bacteria count as it still did not lend itself to depicting significant values related directly to product quality.

Volatile bases showed considerable variations between samples. These variations seemed to originate with the product itself. Frozen breaded shrimp contained at least 3 sources of nitrogen; the batter, the breading, and the shrimp meat. A variation in the content of each one of these components will logically induce a variation in the total nitrogen from which the volatile bases were evolved. In fact, a large percentage of breading should induce a lower total nitrogen in the product and thus lower the amount of volatile bases to be expected, even in a highly decomposed product.

Moisture content of a sample was logically expected to be also a source of variations in the actual per cent volatile bases content.

With these considerations in mind, a ratio of volatile bases to total nitrogen was devised and calculated. It resulted in a decrease in the individual variations found in the data of the two components of the ratio. The ratio of volatile bases to total nitrogen cancelled out the possible variations introduced by the moisture and breading variations because they were determined on the same sample. The curve in Figure 1 illustrates the closest correlation to the average taste panel score. The ratio was seen to increase from an initial value of 6.0 to a value of 7.4 where it remained stationary and then increased to 10.0. At first sight it appeared that this curve could be satisfied by a cubic equation. Mathematical study carried out with the data did not substantiate this conjecture. However, on the basis of the correlation with the average taste panel scores, four zones were depicted (Table 4) which represented four definite degrees of quality in frozen breaded shrimp.

TABLE 4

Ratio values of four depicted zones and their relation to quality in frozen breaded shrimp

Ratio values	Quality
Below 6.0	Very good
Between 6.0 and 7.5	Good
Between 7.5 and 8.5	Questionable
Higher than 8.5	Unacceptable

SUMMARY

Biochemical, organoleptic and bacteriological methods were studied in relation to the development of an objective test for estimating the degree of freshness in frozen breaded shrimp. Moisture and breading content along with total bacteria count, taste panel measurement, total nitrogen and volatile bases were determined on each sample. A quality scale based on the ratio values obtained was proposed to estimate objectively the quality of frozen breaded shrimp. The proposed quality scale was based on chemical tests which could be quickly carried out with a minimum of labor and simple equipment available in any small laboratory. No special technical training is necessary for carrying out this test. For routine testing of relatively homogeneous samples of shrimp, only the volatile bases content need be determined. Total protein content varies in a narrow range. Thus, for plant quality control, VB values furnish valuable qualitative results. However, the acceptability and reliability of this test must be further substantiated by market surveys and by additional plant tests.

Acknowledgment

The authors are indebted to Dr. Rouben Khatchikian (Chas. Pfizer Co., Inc., Brooklyn, N. Y.) for his help in obtaining the bacteriological data.

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Biochemical Methods for Determining Shrimp Quality[a]

II. Survey of Market Frozen Breaded Shrimp[b]

Marcel Gagnon[c] and Carl R. Fellers[d]

Department of Food Technology, University of Massachusetts, Amherst, Massachusetts

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To TEST HOW REALISTIC were the proposed ratio values (VB/TN), presented in a previous paper *(1)*, for determining frozen breaded shrimp quality, analyses were made on samples obtained from retail outlets in 23 U. S. cities. The results were studied for correlation and statistical distribution in regard to actual quality.

METHODS

Samples. The samples of frozen breaded fantail shrimp were shipped from retail stores to the University of Massachusetts in Amherst in insulated containers with dry ice. All samples were solidly frozen upon arrival and were stored at −21.7° C. (−7° F.). The total number of samples analysed amounted to 144 representing 24 brands obtained from 23 (Table 1) United States cities.

TABLE 1

List of United States cities and states from where the survey samples of frozen breaded shrimp were obtained, and the code number assigned

City code No.	City	State
1	Albuquerque	New Mexico
2	Atlanta	Georgia
3	Boston	Massachusetts
4	Chicago	Illinois
5	Dayton	Ohio
6	Denver	Colorado
7	Flint	Michigan
9	Fort Smith	Arkansas
10	Fresno	California
11	Great Falls	Montana
12	Hagerstown	Maryland
13	Hammond	Indiana
14	Kokomo	Indiana
15	Manchester	New Hampshire
16	Milwaukee	Wisconsin
17	Minneapolis	Minnesota
19	New York City	New York
20	Oklahoma City	Oklahoma
23	Portland	Oregon
24	Salt Lake City	Utah
25	San Antonio	Texas
26	Seattle	Washington
27	St. Louis	Missouri

Analytical methods. Using previously described (1) procedures and methods each sample was examined for:

1. Total bacteria count
2. Moisture
3. Per cent breading
4. Total nitrogen (TN)
5. Volatile base (VB)
6. Ratio of VB to TN

RESULTS AND DISCUSSION

Results for the whole survey of the market frozen breaded shrimp are given in Table 2. A study of the correlation established by the data was made by the rank correlation (r_s) method *(2)*.

Moisture was determined in duplicate on the samples and the results were found to vary between 56.5

TABLE 2

Results of bacteriological and chemical tests carried on survey samples of frozen breaded shrimp obtained from a number of U. S. cities

Sample code No.[1]	City code No.[2]	Moisture	Breading	Log of total bacteria per gram	Volatile base calcd. as NH_3 g./100 g.	Total N g./100 g.	Ratio of VB to total N x 100
		%	%				
1 a	24	62.2	59.0	4.85	.150	1.95	7.7
b	24	62.7	58.5	4.91	.130	2.16	6.0
c	17	62.3	53.7	4.41	.143	1.96	7.3
d	17	61.0	43.5	4.54	.138	2.24	6.2
e	12	64.7	61.0	4.33	.108	1.95	5.6
f	19	68.7	52.7	6.22	.126	1.86	6.8
2 a	2	60.6	45.3	4.88	.166	2.50	6.7
b	2	63.4	58.2	5.32	.170	2.70	6.3
c	3	65.5	51.4	4.59	.182	2.10	8.7
d	3	61.4	48.5	5.23	.168	2.24	7.5
e	15	65.3	40.0	5.75	.178	1.97	9.0
f	5	64.1	51.6	5.44	.128	2.48	5.6
3 a	24	63.7	42.3	6.41	.130	1.86	7.0
b	24	64.0	51.5	6.36	.126	1.57	8.0
c	23	58.0	48.0	6.49	.138	1.78	7.7
d	23	64.0	67.6	6.27	.108	1.55	7.0
e	11	65.7	60.0	6.50	.099	1.33	7.5
f	11	66.6	62.1	6.48	.127	2.02	6.1
4 a	5	62.5	46.4	5.75	.133	2.34	5.7
b	5	63.0	48.9	5.44	.130	2.32	5.9
c	1	61.2	46.5	5.47	.140	2.53	5.6
d	20	63.0	46.2	5.37	.150	2.05	7.3
e	17	62.2	42.8	5.47	.123	2.42	5.1
f	6	62.3	34.2	5.94	.145	2.62	5.6
5 a	7	60.7	51.0	6.99	.120	2.04	5.9
b	7	62.6	55.8	6.88	.144	2.16	6.2
c	11	63.2	72.5	6.27	.136	2.46	5.6
d	11	63.2	62.5	4.71	.104	2.04	5.0
e	4	58.4	60.0	6.24	.108	2.03	5.3
f	4	57.5	61.2	6.11	.110	1.97	5.6
6 a	17	61.0	58.0	4.71	.160	1.85	8.6
b	17	61.2	57.5	5.23	.145	1.85	7.8
c	6	63.6	60.7	5.41	.141	2.00	7.1
d	6	62.4	42.8	6.10	.135	1.85	7.3
e	11	62.0	51.7	6.30	.145	1.85	7.9
f	11	62.8	51.3	6.12	.161	2.40	6.7
7 a	25	62.4	65.7	7.08	.136	1.58	8.6
b	25	64.6	55.9	6.86	.135	2.03	6.6
c	13	66.2	58.1	7.05	.126	2.36	5.6

[a] Taken in part from a thesis submitted by Marcel Gagnon to the University of Massachusetts in partial fulfillment of the requirement for the Doctor of Philosophy degree, December 1956.

[b] Contribution No. 1128, Massachusetts Agricultural Experiment Station, Amherst.

[c] Present address: Alphonse Raymond Ltée, Montréal, P. Q., Canada.

[d] Present address: University Laboratories, Amherst, Massachusetts.

TABLE 2 (continued)

Sample code No.[1]	City code No.[2]	Moisture	Breading	Log of total bacteria per gram	Volatile base calcd. as NH_3 g./100 g.	Total N g./100 g.	Ratio of VB to total N x 100
		%	%				
d	13	64.9	51.5	7.30	.110	2.17	5.1
e	1	64.4	57.9	6.04	.117	1.92	6.1
f	6	60.6	57.7	6.67	.139	1.87	7.5
8 a	3	64.7	55.2	5.92	.120	2.07	5.8
b	3	63.4	48.6	5.40	.137	1.89	7.3
c	2	65.7	38.9	6.36	.148	1.65	9.0
d	14	66.0	44.9	6.30	.109	1.66	6.6
e	9	63.7	48.2	5.47	.122	2.04	6.0
f	20	63.8	58.1	6.18	.128	2.42	5.3
9 a	15	64.6	56.4	6.11	.132	1.89	7.0
b	15	64.6	54.2	6.28	.126	1.98	6.4
c	24	66.0	53.6	6.18	.116	2.30	5.1
d	5	69.2	49.0	7.47	.119	1.80	6.6
e	5	66.8	51.0	7.01	.183	2.28	8.0
f	11	59.6	61.7	6.25	.143	1.90	7.5
10 a	16	63.4	44.7	5.72	.134	2.22	6.3
b	16	61.0	47.8	5.15	.112	2.53	4.5
c	19	62.8	51.3	5.55	.159	2.37	7.0
d	19	62.3	37.5	5.73	.122	1.92	6.4
e	4	62.6	39.0	5.18	.134	2.39	5.9
f	13	63.1	40.4	5.14	.154	2.11	7.3
11 a	19	71.8	36.4	7.73	.125	2.07	6.1
b	19	71.2	34.6	6.86	.112	1.87	6.0
c	1	67.6	42.9	7.19	.126	1.93	6.5
d	26	64.0	34.6	5.95	.127	2.15	5.9
e	10	63.8	43.2	6.04	.132	1.98	6.7
f	9	62.2	42.9	5.79	.139	1.89	7.4
12 a	17	67.5	54.3	5.02	.148	1.76	8.5
b	11	67.8	46.3	5.91	.131	1.65	8.0
c	13	65.4	48.5	5.08	.129	1.78	7.2
d	13	64.8	45.7	5.96	.134	2.00	6.7
e	23	66.6	46.8	5.77	.108	1.63	6.6
f	23	63.2	52.8	5.59	.115	1.53	7.5
13 a	13	66.0	48.9	4.56	.111	2.13	5.2
b	13	60.4	57.1	6.12	.129	1.71	7.6
c	4	60.8	55.6	6.28	.132	1.73	7.6
d	2	64.0	58.1	5.89	.104	1.39	7.4
e	14	62.0	51.3	6.05	.139	1.84	7.5
f	9	63.8	52.4	5.76	.128	2.09	6.1
14 a	19	59.8	64.7	5.28	.125	1.35	9.3
b	19	64.8	48.2	5.19	.155	2.32	6.7
c	5	60.0	57.5	5.46	.127	1.57	8.1
d	5	62.6	57.9	5.99	.123	1.70	7.2
e	20	61.2	58.8	5.22	.115	1.43	8.1
f	20	61.0	51.4	5.17	.124	1.35	9.2
15 a	19	56.8	64.4	4.86	.094	1.34	7.0
b	19	61.8	65.9	5.56	.109	1.32	8.3
c	1	61.4	60.7	6.48	.113	1.41	8.0
d	20	57.5	61.7	6.17	.101	1.18	8.6
e	20	58.0	68.9	6.38	.122	1.72	7.1
f	9	57.5	67.2	5.21	.110	1.25	8.8
16 a	27	65.6	39.0	6.06	.136	2.07	6.6
b	27	60.8	47.4	5.97	.146	2.54	5.8
c	25	61.2	55.1	5.62	.161	2.15	7.5
d	10	64.6	46.7	6.16	.135	2.02	6.7
e	26	62.4	40.0	5.74	.131	2.03	6.5
f	7	63.2	44.1	6.15	.124	2.01	6.2
17 a	19	68.9	36.2	5.79	.123	1.71	7.2
b	19	70.1	38.1	5.94	.119	1.59	7.5
c	23	69.3	36.1	6.15	.116	1.66	7.0
d	2	69.4	35.7	5.13	.114	1.49	7.7
e	10	68.0	36.1	5.74	.120	1.83	6.6
f	24	67.5	26.2	5.05	.114	1.82	6.3
101 a	7	64.4	45.7	5.61	.123	1.67	7.5
b	7	57.5	54.3	5.34	.131	1.98	6.6
c	11	63.9	54.5	6.48	.156	1.22	12.8
d	11	62.4	37.5	6.53	.126	1.18	10.7
e	4	61.4	59.3	6.14	.127	1.62	7.9
f	4	63.8	69.0	5.68	.162	2.29	7.1
102 a	2	67.5	40.7	5.35	.144	2.12	6.8
b	2	67.3	43.3	6.76	.146	1.73	8.4
c	19	70.0	31.0	5.32	.142	1.88	7.6
d	23	69.1	42.5	6.00	.135	1.63	8.3
e	10	68.4	36.1	5.65	.146	1.66	8.8
f	24	70.2	50.0	6.71	.151	1.90	8.0
103 a	26	67.8	50.0	5.91	.126	1.71	7.4

TABLE 2 (continued)

Sample code No.[1]	City code No.[2]	Moisture	Breading	Log of total bacteria per gram	Volatile base calcd. as NH_3 g./100 g.	Total N g./100 g.	Ratio of VB to total N x 100
		%	%				
b	26	64.0	48.7	5.99	.100	1.85	5.4
c	24	65.4	50.0	6.27	.125	2.09	6.0
d	24	64.8	43.4	6.49	.110	1.60	6.9
e	23	65.6	41.8	6.12	.127	2.10	6.1
f	11	66.0	50.0	6.25	.140	2.00	7.0
110 a	1	56.5	51.9	5.70	.171	2.12	8.0
b	5	61.0	48.1	5.45	.144	2.13	6.8
c	5	62.7	46.2	5.68	.135	1.83	7.4
d	6	64.4	46.2	5.89	.154	2.12	7.3
e	17	60.0	45.2	5.29	.135	1.95	6.9
f	20	60.4	50.0	5.48	.130	1.65	7.9
113 a	7	64.6	56.2	6.38	.119	1.69	7.1
b	23	65.1	52.6	6.10	.124	1.58	7.9
c	13	64.8	53.2	5.83	.118	1.80	6.6
d	13	65.6	55.0	6.13	.128	1.91	6.7
e	11	63.3	70.3	5.54	.131	1.47	8.9
f	17	64.6	54.3	5.86	.139	1.75	8.0
114 a	17	61.0	52.0	6.03	.131	2.04	6.4
b	17	63.8	58.3	6.09	.126	1.50	8.4
c	6	61.8	47.6	6.19	.125	1.50	8.3
d	6	61.6	58.7	6.08	.114	1.70	6.7
e	11	59.0	58.7	6.12	.151	1.62	9.3
f	11	63.0	57.2	6.12	.146	1.74	8.4
115 a	2	76.0	58.4	6.12	.184	2.41	7.6
b	9	77.5	48.6	6.42	.184	2.98	6.2
c	14	78.3	53.4	5.81	.149	3.03	4.9
d	20	77.5	52.5	5.73	.151	3.06	5.0
e	3	76.9	55.2	5.98	.181	3.04	6.0
f	3	76.1	53.2	5.52	.187	3.04	6.1

[1] Each sample code number represents a brand except for the following which represent the same brand: 3 and 103; 4 and 110; 5 and 101; 6 and 114; 8 and 115; 12 and 113; 17 and 102.

[2] See table 1 for the list of the cities and states corresponding to the code number.

and 78.3%. Average moisture content for all the samples was 64.2%. In the correlation study, no significant relationship was found between the moisture and the total volatile bases content of the samples.

Considerable variations were observed in the breading content of the samples. A maximum of 70.3 and a minimum of 26.2% were found while the average was 50.8%. A negative correlation at the 5% level was obtained between the amount of breading and the total nitrogen indicating that a high percentage of breading in a sample induced a low total nitrogen. On the other hand, no significant correlation was found between the volatile bases, and the percentage of breading.

The logarithm of total bacteria count did not show any significant correlation with volatile bases. Variations encountered in the logarithm of total bacteria count fell within a range of 4.33 to 7.74 with an average value of 5.88. Some samples of good organoleptic and biochemical quality had high counts; others of poor quality had occasional low bacterial counts. These results showed that it was not feasible to use total bacteria count as a criterion of quality in heterogeneous samples of frozen breaded shrimp.

Total nitrogen showed a widespread distribution as was expected from the percentage of breading variations of the samples. The lowest value obtained was 1.18 and the highest 3.06 g./100 g. of frozen breaded shrimp. A strong positive correlation significant at the 1% level was found to exist between the total nitrogen and the volatile bases content of the

frozen breaded shrimp samples. A high total nitrogen content yielded a high concentration of volatile bases.

Volatile base content of the samples had a minimum of 0.094 and a maximum value of 0.187 g./100 g. of frozen breaded shrimp. The fluctuations encountered clearly established that the volatile base determination alone, when made on heterogeneous samples, could be a good qualitative indication of the quality of the sample but would not suffice, by itself, to establish satisfactorily the objective quality of an isolated sample.

Ratio of volatile bases to total nitrogen, multiplied by 100, varied between 4.5 and 12.8. On the basis of the proposed quality scale *(1)* for frozen breaded shrimp, the distribution of the results expressed as percentage are given in Table 3.

TABLE 3

Distribution in per cent of the ratio values (VB/TN) found on survey of frozen breaded shrimp and their relation to the proposed quality zones

Zone values	Samples	Quality
	%	
Below 6.0	17.4	Very good
Between 6.0 and 7.5	47.2	Good
Between 7.5 and 8.5	25.0	Questionable
Higher than 8.5	10.4	Unacceptable

To summarize the characteristics of the frequency distribution of the ratio values of the frozen breaded shrimp samples, the mean (7.12), the mode (6.88), the median (7.04) and the standard deviation (1.21) were obtained. These values were used to determine the degree of skewness (Sk) of the distribution of the ratio values for the entire survey by the following formula *(3)*:

$$Sk = \frac{\text{Mean} - \text{Mode}}{\text{Standard deviation}}$$

The value of skewness should vary between 0 and ± 1. A perfect symmetry or coincidence with the normal distribution curve corresponding would be indicated by an Sk value of 0. A positive value would indicate a tendency of the samples towards a questionable quality on our proposed scale whereas a negative value would indicate a tendency of the samples towards a very good quality.

A postive skewness value of 0.19 was found from the formula, indicating that the distribution of the ratio values was almost symmetrical. The positive value obtained also indicated that the tendency of the samples of frozen breaded shrimp on the market today was towards the questionable quality zone, suggesting that improvements in quality should be undertaken by the industry as a whole to change the skewness from a positive to a more desirable negative value. The skewness value obtained (+0.19) on frozen breaded shrimp demonstrated that the ratio values were realistic as a measure for the degree of freshness. Also the percentage of samples falling within each quality (Table 3) is indicative of what is offered the consumer today.

SUMMARY

This survey of frozen breaded shrimp quality was carried out at the retail store level by means of bacteriological and chemical tests. A total of 144 samples was used. Results showed that the ratio test (VB/TN) was reliable and realistic even in heterogeneous samples. Total nitrogen was found to have the greatest influence on the variations of the volatile base. Moisture and breading content, though playing a part in those variations, could not be found significantly responsible for it. The analysis of the distribution revealed that the distribution of the ratio values was almost symmetrical, thus inferring that the ratio values were reliable. The ratio test can be used as a quality index within the limit of the proposed quality zones for frozen breaded shrimp.

Acknowledgment

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Objective Criteria for Storage Changes in Tomato Paste*

(Manuscript received February 14, 1958)

B. S. Luh, Sherman Leonard and G. L. Marsh

Department of Food Technology, University of California, Davis, California

IN CALIFORNIA, tomatoes are becoming one of the important vegetable crops for processing. During the 1956 season, 2,772,400 tons of tomatoes were processed by California canners and paste manufacturers; this was over 50% of the total tomato crop processed in this country. To save shipping costs, tomatoes for distant markets were to a large extent concentrated and canned as paste, whereas the rest of the crop was canned as juice, puree, catsup, and whole tomatoes.

Factors influencing quality of tomato products have long been a subject of interest to canners and food processors *(15)*. The literature includes reports on the effect of storage temperature on flavor of tomato products *(13)*. Retention of ascorbic acid during storage of tomato paste *(6, 7, 13)* has been extensively investigated.

One of the programs of the Department of Food Technology at Davis is concerned with factors influencing storage stability of processed foods. The interest shown by the growers and canners in the quality of canned tomato products gave impetus to a study of the influence of storage temperature and duration on quality of tomato paste. Several objective criteria for evaluating storage changes in tomato paste are presented.

EXPERIMENTAL

Tomato paste. Pearson tomatoes inspected by the California State Department of Agriculture were received by a California canning company. The fruits were washed, sorted, and trimmed. From the inspection belt the material passed to a chopper and then was pumped immediately into a vertical tubular heat exchanger where it was heated to 93.5° C. (200° F.) in 30 seconds. During this time, tomato polygalacturonase and other enzymes were rapidly inactivated by heat. All contact surfaces were of stainless steel. The product flowed from the top of the heat exchanger to the flashbreak heater, which also acted as a sterilizer and pre-evaporator, where it reached 110° C. (230° F.). Then it moved by gravity to a supply tank serving pulpers and finishers. The hot macerate went to a pulper with an 0.060 in. screen and a finisher with an 0.030 in. screen, which removed seeds, skins, and cores. The juice was concentrated in Buflovak double-effect vacuum evaporators to 26% total solids. The paste was discharged through a tubular heat exchanger where its temperature was raised to 92.2° C. (198° F.). The product was filled into 6-oz. cans with a headspace of ¼ to 5/16 in., which were sealed, processed for 3 minutes at 100° C. (212° F.) in agitating cookers, and cooled in cold water for 7 minutes. The samples were then stored at 6° C. (42.8° F.), 20° C. (68° F.), 25° C. (77° F.), 35° C. (95° F.), 45° C. (113° F.), and 55° C. (131° F.).

ANALYTICAL METHODS

Serum color. Twenty grams of the paste sample was diluted with distilled water to 100 ml. in a volumetric flask. The mixture was centrifuged for 10 minutes. Then 20 ml. of the centrifugate was mixed with 2 ml. of a freshly prepared and filtered solution of 10% Pectinol (w/v) in a 30-ml. Erlenmeyer flask with a glass stopper. The resulting mixture was kept in a water bath at 40° C. for one to two hours to allow hydrolysis of pectic materials. The resulting solution was treated with 0.2 g. of Supercel and filtered through a Whatman No. 2 filter paper. The color of the filtrate was measured in a Klett-Summerson photoelectric colorimeter, in which a blue filter was used. The solution was saved and used in a Folin-Denis test for tannin-like substances. A blank was run, using 20 ml. of distilled water instead of the centrifugate.

Hydroxymethyl furfural (HMF). The method for determining hydroxymethyl furfural content employed primarily the same principle as that described by Amerine *(1)*. However, the extraction and analytical procedures were modified as follows: 30 ml. of centrifugate, obtained by diluting 20 g. of paste with distilled water to 100 ml. as described in the serum color section, was treated with 0.2 g. of Supercel. The mixture was filtered through a Whatman No. 2 filter paper in a Buchner funnel under slight suction. Ten ml. of the filtrate was pipetted into a separatory funnel, to which was added 2.5 g. of reagent-grade sodium chloride. The resulting mixture was extracted 3 successive times, each time with 20-ml. portions of reagent-grade anhydrous ethyl ether. The ether extracts were combined in a porcelain evaporating dish, mixed with 1 ml. of distilled water, and allowed to evaporate at room temperature under an air draft until almost dry. The residue was dissolved and diluted to 10 ml. with distilled water in a volumetric flask. Supercel was added, and the solution was filtered through a Whatman No. 1 filter paper. Three ml. of the filtrate was pipetted into a test tube, followed by 3 ml. each of U.S.P. ethyl alcohol (99.9%) and 1% resorcinol in concentrated hydrochloric acid. The contents were mixed well, and the test tube was stoppered and kept in the dark for 30 minutes at room temperature. The reddish-pink color that developed was measured in a Klett-Summerson photoelectric colorimeter, using a green filter (No. 54). The blank was used to adjust the instrument for 100% transmittance. A standard curve was constructed with HMF concentrations varying from 0 to 180 μg. per ml. sample.

Amino nitrogen. The amino nitrogen content of tomato paste was determined by the ninhydrin colorimetric method of Troll and Cannan *(14)* in the following way: 5 ml. of the centrifugate from the serum color determination (20 g. paste diluted to 100 ml.) was diluted with distilled water to 250 ml. in a volumetric flask. Two ml. of the diluted sample was pipetted into a 50-ml. volumetric flask with a glass stopper. To this were added 5 ml. of KCN-pyridine reagent (2 ml. of 0.01 M solution of KCN diluted to 100 ml. with ammonia-free pyridine, which was prepared by shaking 100 ml. of pyridine with 1 g. of permutite for about 20 minutes) and 5 ml. of 80% phenol solution (80 g. of phenol dissolved in 20 ml. of absolute alcohol and shaken with 1 g. of permutite for 20 minutes). The flask was stoppered and heated in a boiling-water bath for 3 minutes. After partial cooling, 1 ml. of 5% ninhydrin solution in absolute alcohol (w/v) was pipetted into the mixture. The mixture was heated in a boiling-water bath for exactly 5 minutes, counting from the time the water started to boil. The solution was cooled in a cold-water bath and made up to volume

* Presented at the Seventeenth Annual Meeting of the Institute of Food Technologists, Pittsburgh, Pennsylvania, May 15, 1957.

with 60% alcohol. The optical density of the resulting deep blue solution was measured in a Klett-Summerson photoelectric colorimeter, using a green filter. A reagent blank was run, using distilled water instead of the sample. A standard curve was constructed, using glutamic acid concentrations up to 150 μg. per ml. of sample. The results were expressed as mg. amino nitrogen per 100 g. of paste.

Folin-Denis test. The tannin-like substances in tomato paste were determined by the Folin-Denis reagent as described previously *(9)*. Five ml. of the solution from the serum color determination was diluted with distilled water to 50 ml. in a volumetric flask. To 5 ml. of the diluted sample were added 50 ml. of distilled water, 5 ml. of Folin-Denis reagent, 20 ml. of 1 M Na_2CO_3 solution, and water to make 100 ml. The blue color that developed after 1½ hours was measured in a Klett-Summerson photoelectric colorimeter, using a No. 66 red filter. Sometimes it is necessary to centrifuge for 5 minutes to remove the white precipitate. A standard curve for C.P. tannic acid was constructed, with concentrations ranging from 0.05 to 1.0 mg. of tannic acid per 100 ml. of reaction mixture. The results were reported as mg. tannic acid per 100 g. of paste. Correction for interference due to ascorbic acid (1 mg. ascorbic acid is equivalent to 0.8 mg. of tannin-like substances) was made *(9)*.

Ascorbic acid. Ascorbic acid was determined by the 2,6-dichlorophenol indophenol dye titration method *(2)*. Twenty g. of tomato paste was mixed with 50 ml. of 8% acetic acid in water and then diluted to 100 ml. with distilled water in a volumetric flask. The mixture was filtered through Whatman No. 1 filter paper. Five ml. of the filtrate was mixed with 50 ml. of distilled water and titrated with 0.025% 2,6-dichlorophenol indophenol dye until the pink color that developed lasted for 15 seconds. The titer was standardized against C.P. ascorbic acid. Results were calculated as mg. ascorbic acid per 100 g. paste and then expressed as the percentage retained of the amount originally present in the paste.

RESULTS

Effects of storage temperature and time on the chemical and physical properties of tomato paste are rather striking. In general, higher storage temperature causes more rapid deterioration and greatly shortens the shelf-life of the paste.

Darkening of serum color. When tomato paste samples were stored at high temperatures for a certain period of time, the color of the serum changed rapidly from light yellow to deep brown. The degree of darkening can be measured objectively after diluting the paste with water to the concentration of juice (20 g. of 26% solid paste diluted to 100 ml.). Prior to color measurement, clarification of the diluted sample with Pectinol was necessary to remove turbidity caused by soluble pectin. The color of the clarified and filtered serum can be measured in a Klett-Summmerson photoelectric colorimeter. The effect of storage temperature and time on diluted serum color of tomato paste is shown in Figure 1.

The sample stored at 6° C. (42.8° F.) for 448 days showed practically no darkening of serum color. Sera from samples stored for the same period at 20° C. (68° F.) and 25° C. (77° F.) darkened only slightly. Sera from samples stored at 35° C. (95° F.), 45° C. (113° F.), and 55° C. (131° F.) darkened in a very short time. The rate and extent of darkening increased as storage temperature rose. Serum color might therefore be used as a test for tomato paste quality. Samples with darkened serum color invariably indicate a poor storage condition, i.e., storage at high temperatures for a long period of time.

Hydroxymethyl furfural content (HMF). Hydroxymethyl furfural can be formed by heating keto-hexose sugars in acid solution *(1)*. When tomato paste is stored at higher temperatures, the formation of HMF from reducing sugars through acid catalysis or browning reaction *(5)* is possible. Results of the effect of storage temperature on HMF content of tomato paste are shown in Figure 2. At higher storage temperatures, HMF content was found to increase rapidly as the storage period lengthened. However, no significant increase was found

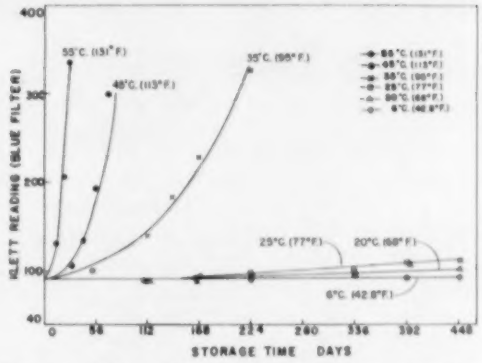


Figure 1. Effect of storage temperature on serum color of canned tomato paste.

among the samples stored at 6° C. (42.8° F.), 20° C. (68° F.), and 25° C. (77° F.). Thus, HMF formation is dependent on the storage temperature. To avoid HMF formation storage temperatures of 25° C. (77° F.) or lower are more desirable. The quantity of HMF present in a given tomato paste sample might be used as an objective criterion for evaluating quality. A high HMF content in the sample indicates that it has been subjected to high storage temperature for a period long enough to cause chemical deterioration.

Decrease in amino nitrogen. Amino nitrogen content of tomato paste decreased as the storage period lengthened. The rate of decrease was especially rapid at high storage temperatures. Figure 3 indicates the trends of such changes. Loss of amino nitrogen at 6° C. (42.8° F.) was practically nil even after 448 days. The decrease in amino nitrogen content during storage may be considered as undesirable in regard to nutritive value and flavor acceptance. This should be avoided by storing the canned paste at low temperature. The rapid decrease in amino nitrogen content of tomato paste stored at high temperatures might be related to the Maillard reaction—a chemical reaction between amino groups in proteins, peptides, and amino acids and the carbonyl groups in reducing sugars *(5)*. This phenomenon may also be related in part to darkening of serum color as shown above.

Folin-Denis test. Storage changes in tomato paste may also be measured by the Folin-Denis test for tannin-like substances containing aromatic hydroxyl groups. A deep blue color formed by reduction of the tungstate and molybdate in alkaline solution is a measure of the amount of tannin-like substances. Since ascorbic acid also reacts with this reagent to give a blue color, correction for the interference due to ascorbic acid was made. Results shown in Figure 4 indicate a gradual increase

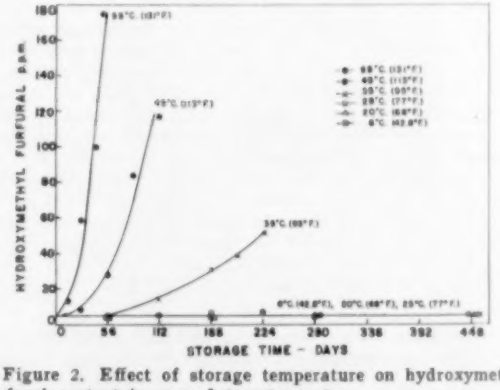
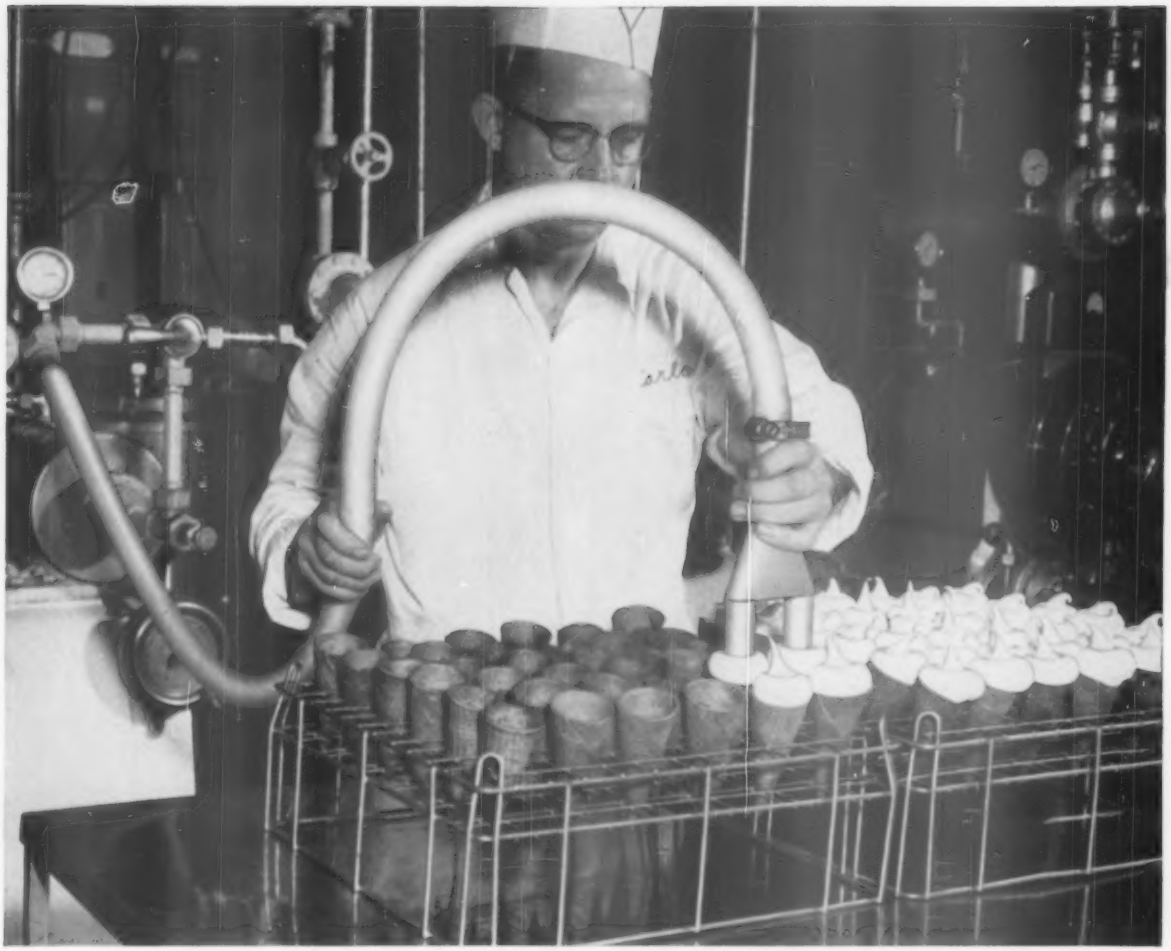


Figure 2. Effect of storage temperature on hydroxymethyl furfural content in canned tomato paste.



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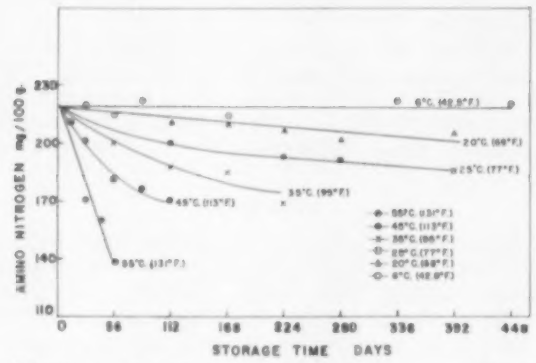

Figure 3. Decrease in amino nitrogen in canned tomato paste stored at different temperatures.

in tannin-like substances with storage. Thus, a paste sample with a high Folin-Denis test is an indication of severe storage conditions.

Decrease in ascorbic acid. Results in Figure 5 show the effect of storage temperature and time on ascorbic acid retention in tomato paste. Before storage, the ascorbic acid content of the paste was 78.0 mg./100 g. Ascorbic acid decreased far more rapidly at higher temperatures. With respect to nutrition, storage at 6° C. (42.8° F.) was far better than the higher temperatures. Storage temperatures above 25° C. (77° F.) caused more rapid decrease in ascorbic acid and shortened the shelf-life greatly. In the set of samples studied in this experiment, a 50% reduction of ascorbic acid is reached in 82 days at 35° C. (95° F.), in 32 days at 45° C. (113° F.), and in 13 days at 55° C. (131° F.). In the low storage temperature range, ascorbic acid content seemed to be more indicative for quality evaluation than the serum color or HMF method because of the pronounced difference in ascorbic acid content among the samples stored at 6° C. (42.8° F.), 20° C. (68° F.), and 25° C. (77° F.) for 448 days.

Decrease in vacuum. Tomato pastes stored at high temperatures tend to lose vacuum rapidly; at 55° C. (131° F.), the vacuum reading decreased from 9 in. to zero within 42 days. At 45° C. (113° F.), the vacuum reading decreased to zero within 84 days. At 6° C. (42.8° F.), 20° C. (68° F.), and 25° C. (77° F.), the vacuum readings of the canned paste samples were still above 5 inches even after storage for 448 days. At the later stage of high temperature storage, the can ends bulged. This might be explained by the decrease in oxygen, evolution of hydrogen resulting from reaction of iron of the can with the contents of tomato paste after the tin coating is dissolved, and possibly to evolution of carbon dioxide gas caused by the browning reaction of the reducing sugars with amino acids and proteins. Thus, a low vacuum in canned tomato paste might be indicative of a poor storage condition and inferior quality.

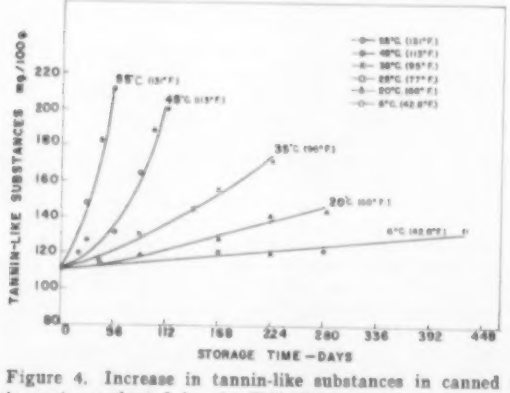

Figure 4. Increase in tannin-like substances in canned tomato paste, evaluated by the Folin-Denis test.

DISCUSSION

The standard of identity for canned tomato paste *(16)* requires a minimum of 25% by weight of salt-free tomato solids. In addition, color and absence of defects are considered in grading the canned product. To meet the Grade A Fancy requirement, the paste must have a color score of not less than 51 points out of a maximum score of 60 points, obtained by comparing the color of the product with that produced by spinning a combination of Munsell color discs *(16)*. However, undesirable flavor and chemical changes in stored tomato paste may proceed in improperly stored tomato paste and may not be detected easily by the color score method. This is because the red color of tomato paste comes mainly from lycopene and other carotenoids which are more stable toward adverse storage temperature than the flavor constituents. Darkening of the serum of the paste could also impair the color of tomato paste.

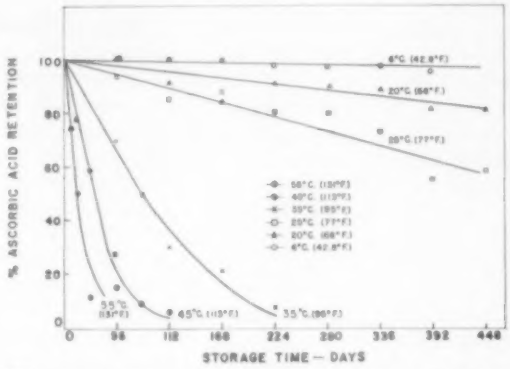

Figure 5. Changes in ascorbic acid content of canned tomato pastes stored at different temperatures.

However, because of the dominant red color of lycopene and carotenoid present in the sample, brown-colored pigments have less influence on the color grade unless the storage temperature is unusually high. The results described herein on changes in serum color, hydroxymethyl furfural, and ascorbic acid content might be used to supplement the color test in evaluating tomato paste quality as they are more sensitive toward storage conditions. The data presented herein, however, show only trends of such changes. More information is needed before standards can be established to evaluate quality of tomato paste in terms of chemical and physical properties.

The change in serum color during storage might be explained by the formation of unsaturated color polymers of varying composition *(5, 11, 12)*. This occurs most commonly through the Maillard reaction, a chemical combination between amino and carbonyl groups *(5, 8, 10)*, and the caramelization process—a chemical reaction between sugars (polyhydroxycar-

bonyl compounds) and polyhydroxycarboxylic acids *(5, 11, 12)*. The Amadori rearrangement of N-substituted glycosylamines, the dehydration and fragmentation of sugar moiety to furfural and other carbonyl compounds, and the formation of hydroxymethyl furfural from amino-carbonyl reaction might explain in part the browning of serum color *(5)*. Furthermore, degradation of ascorbic acid to furfural and carbon dioxide was likely related to the browning reaction, because ascorbic acid contents decreased as the serum color increased. Thus, many reactions, all of which were accelerated by high storage temperature, although at different rates, could contribute to the change in serum color. The extent of such storage changes increases with the lengthening of storage time. Since tomato paste samples with high serum color were invariably found to be poor in flavor, every effort should be made to retard such changes by keeping storage temperature low, preferably below 25° C. (77° F.).

Hydroxymethyl furfural (HMF) can be formed by heating keto-hexose sugars in acid solution *(1)*; it can also result from the glucose-amino acid browning reactions *(5)*. The tomato paste used in this study had an acid content of 1.4 to 1.6% as citrate acid, which may cause the formation of HMF at high storage temperature by acid catalysis and browning reactions. The samples stored at 6° C. (42.8° F.), 20° C. (68° F.), and 25° C. (77° F.) did not show any significant increase in HMF content even after storage for 448 days, while those stored at higher temperatures showed a rapid increase even after short storage periods. The presence of HMF in tomato paste is an indication of poor storage condition and quality, and might be used as a criterion for tomato paste quality.

Amino nitrogen content in tomato paste is important to flavor acceptance and to the biological availability of the proteins and amino acids. Results obtained in our laboratory have indicated that glutamic acid is one of the major amino acids in tomato fruit. The loss of amino nitrogen in tomato paste during storage might be caused largely by the carbonyl-amino type of condensation reaction *(5, 8)*, which also contributed to darkening of the serum color. Undesirable colored compounds may be formed by the browning reaction and cause poor flavor acceptance of the product.

Ascorbic acid content has been used as a criterion of storage changes in tomato juices *(3, 4, 6, 7, 13)* and paste *(6, 7, 13)*. Results obtained in our experiment agreed reasonably well with those appearing in the literature *(7, 13)*. In Figure 5, more rapid destruction of ascorbic acid in tomato paste stored at higher temperatures is shown. Because ascorbic acid content in tomatoes and tomato products varies with variety, climate, and processing methods, it is difficult to determine the previous storage condition of tomato paste by ascorbic acid content alone. However, for quality evaluation in a cannery, the test is of value to study storage changes.

Solution of tin and iron from the can into the paste during storage could also influence flavor. Although these constituents were not investigated in this work, they surely may play an important role in quality changes.

Results obtained in this work indicate that at high temperatures tomato paste deteriorates rapidly. Storage at 25° C. (77° F.) or lower seems to be preferable for quality retention. This agrees with previous studies on tomato concentrates *(13)*.

SUMMARY

Tomato paste made by the hot-break process and containing 26% total solids was stored at temperatures varying from 6° C. (42.8° F.) to 55° C. (131° F.) for 15 months. The samples were tested for changes in chemical constituents. Results indicate that tomato paste deteriorates rapidly at temperatures higher than 25° C. (77° F.). Serum color was found to darken, and increases in hydroxymethyl furfural and tannin-like substances occurred. High storage temperature and a long storage period also contributed to decreases in amino nitrogen, ascorbic acid, and vacuum. Among these, determination of serum color and hydroxymethyl furfural content were found to be promising as objective criteria for evaluating quality of tomato paste. To maintain quality, storage at low temperature, preferably below 25° C. (77° F.), is desirable. The lower the storage temperature, the slower the rate of chemical deterioration. The objective tests might be useful as supplements to the prevailing methods of grading tomato paste. The effect of storage temperature on shelf-life and chemical composition of tomato paste is discussed.

Acknowledgment

The authors wish to thank Prof. E. M. Mrak for his guidance and interest in this work. The technical assistance of Mrs. Violet Lew is gratefully acknowledged.

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Quality Evaluation of Deep-Fat Fried Peas[a]

Wattana Sathiraswasti[b]
Department of Horticulture, Kasetsart University, Bangkok, Thailand
and
D. K. Salunkhe
Department of Horticulture, Utah State University, Logan, Utah

(Manuscript received December 17, 1957)

Dehydration is perhaps the oldest method of preserving fruits and vegetables. It is still an important method of preservation. During World War II, efforts were made to determine the potentials of certain newer dehydrated foods such as powdered eggs, tomatoes, and potato granules. Since then, many new products have been coming into the market every year. The use of dehydrated products has increased primarily because they usually require less space and have longer shelf life. At the same time they have a much more concentrated nutritive value than fresh, canned, or frozen foods. Hence, they are adapted to use in military feeding. Because of their ease of handling and transportation, they may also serve as a snack or picnic food *(4, 8)* in civilian feeding.

Dehydration of fruits and vegetables by deep-fat frying is a comparatively new method. A deep-fat fried product not only has a longer shelf life, but also an additional nutritional ingredient, fat.

For most of the countries of Asia, it seems to the authors that the preservation of fruits and vegetables by deep-fat frying is both an appropriate and an adequate method because it is simple and cheap and at the same time the deep-fat fried product, for example, peas, is rich in protein and carbohydrate. By addition of fat it can be used as a nutritious food by large masses of vegetarians.

Over-mature peas of size 6 bring substantially less income to farmers than those of size groups 1–3 or 4 because the latter are usually the choice peas for canning and freezing. By deep-fat frying of peas of sizes 5 and 6, the crop could be better utilized. This means additional income to farmers who grow peas for the canning industry *(12)*.

The investigations presented in this paper comprise the evaluation of certain objective methods such as size distribution, shear-press, specific gravity, refractive index, and starch grain character (size, shape, and hylum) in relation to the harvest time and the quality (organoleptic appraisal, color, moisture, protein, and fat) of the deep-fat fried peas.

EXPERIMENTAL

Four varieties of peas: Giant Stride, Little Marvel, New Era, and Victory Freezer were planted on the Terry Experimental Farm, Logan, Utah, on approximately ⅛ acre on May 2, 1957.

Peas were harvested at 3 different intervals. The time interval between harvest was not uniform. The harvest date began July 8 and ended on July 29, 1957.

Subsequent to harvest, the shelled peas were utilized for the following studies:

1. Objective measures for maturity.
2. Development of the deep-fat frying technique.
3. Determination of nutritive value and color changes in deep-fat fried peas.
4. Organoleptic appraisal of deep-fat fried peas.

1. Objective measures for the maturity of peas

Certain objective measures were made to determine the ma-

[a] This paper is an abstract of the thesis submitted by the senior author in partial fulfillment of the degree of Master of Science in Horticulture at the Utah State University, December, 1957. Contribution No. 53 from Utah State Agricultural Experiment Station, Logan, Utah.

[b] Rockefeller Foundation Fellow (1956–57) from Kasetsart University, Bangkok, Thailand, to Utah State University, Logan, Utah, U.S.A.

PLAN OF HARVEST

Variety	1st Harvest	2nd Harvest	3rd Harvest
Giant Stride	July 26	July 28	July 29
Little Marvel	July 8	July 9	July 10
New Era	July 13	July 17	July 21
Victory Freezer	July 19	July 22	July 24

turity of peas on the different harvest dates and to correlate them with the quality of the deep-fat fried peas. They were:

Size distribution. The shelled peas were classified into 4 different size groups, as follows: Sizes 1–3 were those which could be screened through a sieve 5/16 in. Size 4 was the designation of peas which could be screened through a sieve 6/16 in. but were held on a 5/16 in. sieve. Size group 5 was the designation given peas which screened through a sieve 7/16 in. but were held on a 6/16 in. sieve. Peas of size 6 were those which were held on a 7/16 in. sieve.

Specific gravity. The specific gravity determination of peas was conducted according to the method described by Lee *(5)*.

Shear-press values. Tenderness was measured by the Shear-press instrument.[3]

Refractive index values. Expressed juice of the peas from the Shear-press instrument was used in determining the per cent of soluble solids, mostly sugars by Abbe type refractometer *(2)*.

Microscopic examination of starch grains. Random samples of peas from each size group were thoroughly cleaned with water after removing the shriveled peas. Peas of each size group were preserved in 95% ethyl alcohol. Slurries of these samples were prepared with a Waring blender.

Pea slurries were used for the microscopic examination of the starch grains. The size, shape, and hylum[4] of starch grains were observed under the 44 x objective lens and a 10 x ocular.

2. Development of the deep-fat frying technique

A 500-g. sample of peas from each size group was fried in a Westinghouse electric pan with 5000 c.c. capacity. "Dri-Fri" cocoanut oil was used.

Dri-Fri was heated to 350° F. and used with both blanched and unblanched peas. Each pea sample was put in the strainer and dipped in boiling (350° F.) oil. A quick dipping technique was developed that caused popping of the peas and resulted in a superior product. The sample in the strainer was well covered before putting it into the hot oil. The peas were promptly dipped into the boiling oil for a few seconds. Consecutive dippings were made until the sizzling subsided. The cover was then removed, and the peas allowed to fry 12–13½ minutes longer. When the bubbles stopped in the oil, the cooking was discontinued.

3. Determination of nutritive value and color changes in deep-fat fried peas

In evaluating the nutritive values of the product, the following chemical analyses were conducted:

Protein. The Kjeldahl method for total nitrogen determination was employed. The per cent of total nitrogen was computed and converted into crude protein by multiplying by a factor of 6.25.

Fat. Fat was determined by the Ether extraction method.

Moisture. A known weight of ground deep-fat fried pea sample from each size group was dehydrated in a drying oven at 158° F. for 24 hours, then transferred to a vacuum oven at 158° F. at 20 pounds reduced pressure for 48 hours.

Color and color difference. The color of the ground deep-fat fried peas was determined by the Hunter color and color difference meter.[3] The kitchen green panel, a color standard (Rd 34.0, a − 12.2, b 15.3) was employed for standardization of the readings. The Rd color scale was set in the experiment. The ground peas were put in a clean petri-dish and color reflectance was measured. Rd, a, and b values were recorded, totaling 4 readings on each sample. They were interpreted as follows:

Rd value—luminous reflectance
a minus value—greenness
b value—yellowness

4. Organoleptic appraisal of deep-fat fried peas

Organoleptic appraisal was conducted to determine the acceptability of deep-fat fried peas. A trained panel of 10 judges was used. The hedonic scale *(7)* was employed by the judges to express their reaction to the samples. A score of 9 indicated a *like extremely* and a score of 1 indicated a *dislike extremely*. A sample receiving a score of 5 or more was considered to be acceptable.

The comparison between the fried products from blanched and unblanched peas was made to determine the difference of the quality and flavor of the final product.

Data obtained in these investigations were analyzed for statistical significance *(6)*.

RESULTS AND DISCUSSION

Size distribution. In general, the yield of shelled peas in each of the size groups increased as the harvesting was advanced.

Shear press. Table 1 indicates the variability in shear-press values in different sizes, harvest dates, and varieties. In each case, the peas of different size groups markedly influenced the shear-press values. That is, peas of size 6 showed higher shear-press values than those of 1–3, 4, and 5 size groups.

Refractive index. Table 1 shows that in all 4 varieties, the refractive index values decreased as the sizes of peas increased from size group 4. The data showed that the highest per cent of refractive index values were in peas of size 4. That is, the sweetest peas were from the sieve size 4. This is in agreement with previous work *(12)*.

Generally, in the processing industry, peas of size 1–3 bring a higher price to the grower than those of size 4. This is because the peas of size 1–3 are more tender than the peas of size 4. However, peas of size 4 contain more sugars than peas of size 1–3, 5, or 6. (Table 1). Hence, they usually have a better flavor than the peas of size 1–3, 5, or 6. It is, therefore, recommended that the grower should receive equal amount of money for his peas of size 1–3 (tender) and 4 (sweet). This can be confirmed with shear-press and refractometer readings. It is also recommended that the peas of size 1–3 and 4 may be utilized for canning and freezing; peas of size 5 may be canned as second grade peas; and the peas of size 6 (starchy) can be utilized by deep-fat frying. By adapting this practice, the grower will receive a better share of the consumer's dollar from the processor than what he received today.

Specific gravity. Table 1 shows that the specific gravity of peas of different sizes was highly influenced

[3] Bridge Food Machinery Co., Philadelphia 35, Pennsylvania *(3)*.

[4] The term hilum means the mark at the point of attachment of an ovule to its funicle, forming the eye as in the bean. In addition, hilum has another meaning, i.e., the nucleus of a starch grain. To differentiate these meanings Salunkhe and Pollard *(9, 10, 11, 12)* coined the word *hylum*. In this paper, hereafter, the word hylum (plural hyla) will be employed to designate the nucleus of a starch grain.

[3] Manufactured by Henry A. Gardner Laboratory, Inc., Bethesda, Maryland *(1)*.

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TABLE 1

Effect of harvest times and sizes of peas on the average shear-press, refractive index, specific gravity, and starch grain size of peas of four varieties

Objective methods	Varieties				Harvests			Size groups			
	Giant Stride	Little Marvel	New Era	Victory Freezer	I	II	III	1–3	4	5	6
Shear press	971.0	710.0	753.3	750.8	738.8	805.1	845.0	569.0	719.2	854.2	995.4
Refractive index (% soluble solids)	13.32	14.60	14.73	13.49	14.50	14.02	13.59	14.13	15.16	14.18	12.68
Specific gravity	1.0657	1.0412	1.0540	1.0548	1.0460	1.0564	1.0590	1.0433	1.0524	1.0579	1.0620
Starch grain size (microns)	16.12	14.26	15.01	14.18	15.46	14.32	14.90	9.36	13.58	16.88	19.77
Unhylumed starch grains, %	35	55	43	44	44	47	42	79	58	27	14

L.S.D. @ 0.05 = Shear press—Varieties and size groups, 37.8; Harvests, 32.7.
= Refractive index—Varieties and size groups, 0.79; Harvests, 0.68.
= Specific gravity—Varieties and size groups, 0.0013; Harvests, 0.0011.
= Starch grain size—Varieties and size groups, 1.75; Harvests, not significant.
= Unhylumed starch grains—Varieties and size groups, 8; Harvests, not significant.

by both size groups, varieties, and harvest dates.

Starch grain characters. *Starch grain size:* Table 1 shows that the size of starch grains (in microns) was increased as peas increased in size.

Starch grain shape. It was noticed that the varieties of peas differ in shape of starch grains but harvest dates and size groups of the peas showed no significant variations. The circular shape of starch grains was rather common. Beside this, the rectangular and the polygonal shapes were noticed in the minority.

Structural development of hylum. It was concluded from the voluminous data (Table 1) that, as the peas increased in size, the structural development and ramifications of the hylum increased. This may be the result of the progressive dehydration of peas as they mature *(10, 11, 12)*.

Deep-fat frying. If the immersion temperature is below 350° F., it influences the popping action (breaking of testa) of peas. The exit temperature should be kept at the appropriate level depending upon the pea sizes. If the immersion temperature is allowed to increase beyond 350° F., the peas will burn. The time required for frying, which depends upon the sizes of peas (Table 2), should also be strictly adhered to; otherwise, the flavor will be altered.

The amount of weight lost by the peas was inversely related to the size of peas. However, the small peas retained more moisture and showed greater loss of weight. When peas were immersed in oil at 350° F., they were a tasty and "crunchy" product.

Previous work on deep-fat frying of peas by Salunkhe, Pollard, and Taylor *(12)* suggested a temperature of 300° F. for frying over-mature peas.

TABLE 2

Temperature changes and time required for frying 500 grams of peas and the weight of fried peas (average of four varieties and three harvest dates)

Size groups	Immersion temperature	Temperature immediately after immersion	Exit temperature	Time required for frying	Weight of fried peas
	°F.	°F.	°F.	*Minutes*	*Grams*
1–3	350	220	280	13.0	120
4	350	220	290	13.5	140
5	350	225	300	14.0	170
6	350	235	310	14.5	180

Kelley and Baum *(4)*, however, advised the use of a temperature of 270–300° F., for deep-fat processing of carrots, beets, parsnips, peas, and lima beans. Findings of these workers did not agree with the findings of the present authors with regard to the temperature used. After extensive experimentation on deep-fat fried peas, it was found that the popping action is dependent upon 3 main factors:

1. The immersion temperature must not be less than 350° F. If the temperature is below 350° F., it will be hot enough to break the seed coat of the peas.

2. The dipping technique (discussed under Experimental).

3. The uniform ratio of peas and oil (in this experiment, a ratio of 500 g. of peas to 2800 c.c. of cocoanut oil).

Blanching did not have any significant effect on the popping action of peas. This agrees with the earlier work of Kelley and Baum *(4)*.

Nutritive value. Differences in protein content between peas of different sizes and varieties were highly significant (Table 3). Over-mature peas (size groups 5 and 6) yielded the product of a higher protein content than from immature peas (size groups 1–3 and 4).

Table 3 shows that there was an inverse relationship between the size of peas and the moisture content of the final product. The harvest dates and varieties had no effect on the moisture content of the deep-fat fried peas.

It is evident from Table 3 that the fat content of the product was inversely proportional to the size of the peas, but harvest dates and varieties showed no significant variations in the fat content of the product.

Color and color difference. Table 4 shows that the Rd values (luminous reflectance) obtained on ground deep-fat fried peas were highly influenced by the size of peas. The varietal difference had some effect (at 5% level) but harvest dates showed no effect on Rd values. The product of small size peas was less shiny (reflection of light) than that of the large size.

Table 4 shows that the size of peas and varieties changed the "a" values (greenness) of the products. Harvest dates failed to show any effect on the "a" values. The fried product made from young peas was

TABLE 3

Effect of harvest times and size of peas on the average per cent protein, fat, and moisture contents of deep-fat fried peas of four varieties

Nutritive ingredients, %	Varieties				Harvests			Size groups			
	Giant Stride	Little Marvel	New Era	Victory Freezer	I	II	III	1–3	4	5	6
Protein	18.52	17.58	19.50	19.58	18.37	19.02	18.99	17.36	18.45	19.33	20.04
Fat	25.67	28.03	26.57	27.19	28.03	26.47	26.10	31.09	26.96	25.02	24.40
Moisture	3.14	3.24	3.19	3.37	3.16	3.36	3.18	3.72	3.35	3.07	2.79

L.S.D. @ 0.05 = Protein—Varieties and size groups, 0.90; Harvests, not significant.
= Fat—Varieties and size groups, 2.00; Harvests, not significant.
= Moisture—Varieties and size groups, 0.20; Harvests, not significant.

TABLE 4

Effect of harvest times, sizes of peas on the average Rd, a, and b values obtained by Hunter color and color difference meter on ground deep-fat fried peas of four varieties

Hunter color values	Varieties				Harvests			Size groups			
	Giant Stride	Little Marvel	New Era	Victory Freezer	I	II	III	1–3	4	5	6
Rd	13.76	11.86	13.62	12.82	12.55	13.13	13.36	10.54	11.86	14.30	15.35
a	−38.36	−39.76	−36.97	−39.60	−38.37	−38.95	−38.72	−42.60	−39.32	−37.14	−35.65
b	22.05	21.05	22.03	21.62	21.50	21.71	21.85	19.98	21.02	22.67	23.07

L.S.D. @ 0.05 = Rd—Varieties and size groups, 1.40; Harvests, not significant.
= a—Varieties and size groups, 1.86; Harvests, not significant.
= b—Varieties and size groups, 0.78; Harvests, not significant.

greener and browner than the product obtained from over-mature peas.

Table 4 indicates that size groups of peas influenced the "b" values (yellowness) of the product. Varieties of peas had some effect (5% level) but harvest dates had no effect on the "b" values of the product. The deep-fat fried product obtained from the large size peas (5 and 6) was more yellow than those prepared from the smaller size (1–3 and 4) peas.

The color of the product is one of the important factors in consumer acceptance. The dark green color of the deep-fat fried peas is not highly appealing. An attempt, therefore, was made to "dip" the deep-fat fried peas in chocolate to add chocolate flavor and to make an attractive product.

Organoleptic appraisal. Table 5 indicates that the product from the large size peas was better in quality and in flavor than that from small size peas. The quality and flavor of the product depends mainly upon the size and varieties of peas, but harvest dates did not show any effect on the quality.

Regardless of varieties and harvest time of peas studied, it may be concluded, in general, that the taste quality of deep-fat fried peas seems to be directly correlated with the size, shear-press values, specific gravity, starch grain size, and the structural development of hylum of starch grains of peas (Figure 1).

It may be further concluded that the harvest dates studied did not show significant effects, as a rule, on the objective measures for the determination of the potential quality in raw peas or the final quality of deep-fat fried peas. This may be attributed to the following reasons:

The varieties of peas used in this experiment set pods indeterminately

The interval between harvests may be too short.

The results from the comparison of blanched vs. unblanched did not show any difference in the flavor and quality. The blanching of peas was assumed to be unnecessary and hence the treatment was discontinued in further experiments.

SUMMARY AND CONCLUSIONS

Four varieties of peas—Giant Stride, Little Marvel, New Era, and Victory Freezer—were planted at the Terry Experimental Farm, Logan, Utah on May 2, 1957; and harvested from July 8 to July 29. Various objective tests were conducted to determine the proper maturity of peas for deep-fat frying. The process of deep-fat frying was developed. Chemical analyses were conducted to determine the nutritive value of the product. Organoleptic appraisal was also arranged.

Objective tests. Peas were classified according to

TABLE 5

Effect of harvest dates and size of peas on the average organoleptic appraisal scores on deep-fat fried peas of four varieties

Varieties				Harvests			Size groups			
Giant Stride	Little Marvel	New Era	Victory Freezer	I	II	III	1–3	4	5	6
7.0	7.5	7.3	6.9	7.3	7.1	7.1	6.6	7.0	7.4	7.7

L.S.D. @ 0.05 = Varieties and size groups, 0.2; Harvests, not significant.

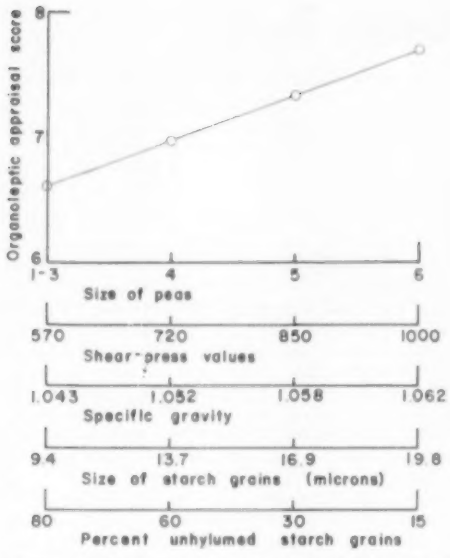

Figure 1. Relationship between organoleptic appraisal score for deep-fat fried peas and size, shear-press value, specific gravity, size of starch grains, and per cent unhylumed starch grains of raw peas.

the size groups before initiating the objective tests. The results were:

Shear-press values, specific gravity, size of starch grains and structural development of hylum of starch grains increased as the size of peas increased, whereas the refractive index values increased at first and then decreased abruptly as the size of peas increased. Different shapes of starch grains of peas were also observed.

Deep-fat frying. Peas were fried in cocoanut oil (Dri-Fri) at 350° F. Proper frying technique was developed for the experiments.

Quality of deep-fat fried peas. The protein content of deep-fat fried peas increased whereas fat and moisture contents decreased as the size of peas increased.

The over-mature peas (size 6) when deep-fat fried had better "eye appealing" color than that of immature deep-fat fried peas.

The taste of the deep-fat fried peas was directly correlated with the size of raw peas.

The harvest dates did not show significant effect on the objective measures for the determination of the potential quality in raw peas as well as the final quality of deep-fat fried peas. This may be attributed to the indeterminate pod set and the short interval between harvests.

The findings of this study could be helpful to the canning industry since over-mature peas are of little value for processing purposes. If the over-mature peas were utilized by deep-fat frying, the processors as well as the growers of peas would benefit from the additional income.

Acknowledgments

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Observations on the Consistency of Tomato Paste

C. J. B. Smit and B. K. Nortje

Food Technology Section, W. P. Fruit Research Station, Stellenbosch, Union of South Africa

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SINCE CONSISTENCY is one of the major factors determining the quality of tomato juice, purée and paste, a considerable amount of work has been done to establish the factors responsible for a good consistency in such products. Kertesz and Loconti *(5)* found that a high serum viscosity, mainly due to the presence of pectic substances, increased the gross viscosity of canned tomato juice. A high pectin content could however only be obtained in cases where the fruit was rapidly heated to a sufficiently high temperature after breaking in order to stop enzymic breakdown. McColloch and Kertesz *(7)* stated that the loss of pectinic substances due to enzyme action was probably a result of a demethylation of pectinic acid by pectin methylesterase, followed by a depolymerization of pectic acid to polygalacturonic acid units catalysed by pectic acid depolymerase. The need for rapid heat inactivation was also demonstrated in the case of tomato paste and purée by McColloch *et al. (8)* and Luh *et al. (6)* who showed that poor consistency resulted from improper heat inactivation of pectic enzymes.

More recent work [Hand *et al. (3)*; Robinson *et al. (9)*; Whittenberger and Nutting *(10)*] established the fact that the quantity and quality of insoluble material present in tomato juice had a very important bearing on consistency and the related property of settling. It was shown that the speed and the screen size of the finisher influenced gross viscosity of the juice very markedly, and that a long holding period between chopping and preheating of tomatoes did not materially affect gross and serum viscosities. In addition, serum viscosity as influenced by pectin content, did not have any direct effect on settling.

In the present study an attempt was made to determine the effect of rapid and delayed preheating of broken Pearson and San Marzano tomatoes on the consistency, comparative serum viscosity, and composition of the final paste.

EXPERIMENTAL

Preparation of tomato paste. During the 1956 and 1957 seasons Pearson and San Marzano tomatoes were obtained from the Cape Flats area and ripened at room temperature if necessary. After thorough mixing, washing and trimming, the fruit of each variety was separated into equal lots for further processing. For breaking, a laboratory-type Chisholm-Ryder tapered screw juice extractor, fitted with a ¼-inch screen, was used. In those cases where no delay between breaking and preheating was allowed, the broken fruit fell directly into a small amount of boiling water held in a steam jacketed kettle. Heating was controlled during breaking to get the temperature to above 180° F. in the shortest possible time. Other lots again were held for 3 hours at 70–80° F. after breaking, before they were heated to 180° F. All the lots were "finished" at approximately 180° F. by passing through the tapered screw juice extractor which was fitted with either a .02 inch or a .04 inch screen.

The different lots of juice were then concentrated in an A.P.V. laboratory-type concentrator at a vacuum of 25–28 inches of mercury, and during concentration samples were frequently taken for consistency determinations. Samples of the original juice and the final paste were also canned and stored at 34° F. until analysed.

To determine the effect of Pectinol treatment, samples of the paste as prepared above were diluted to 15° B., divided into two, and 0.2% Pectinol A added to the one half of each sample. All samples were then left at 40° C. for 24 hours after which they were heated to above 180° F. and cooled.

Consistency determinations. These determinations were carried out with a Bostwick type consistometer [Davis *et al., (2)*]. The dimensions of the chamber were 5 cm. x 5 cm. x 5 cm. and its flowpath was 5 cm. wide, 15 cm. long graduated in 1 mm. divisions. The extent of flow was measured after a definite time interval had elapsed after opening the gate. This time interval was taken to give a good reading without undue serum separation. All determinations were made at 23 ± 1° C., and values reported are averages of at least four readings.

Previous authors [Davies *et al. (2)*; Luh *et al. (6)*] compared various methods for the determination of consistency of tomato paste and showed that the results obtained with different methods are not necessarily comparable. Because it was possible in this work to use the Bostwick consistometer over a fairly wide range of concentrations without previous dilution, this method was used throughout during this investigation.

Preparation of samples for further analysis. Paste samples were diluted to approximately 10° B. with distilled water, heated to boiling point and cooled to room temperature. Total solids and water insoluble solids were determined in this diluted paste. Serum for the determination of relative serum viscosity, calcium pectate and methoxyl contents was obtained by centrifuging the diluted paste at 3500 r.p.m. for 10 minutes, followed by filtration of the supernatant on Whatman No. 54 paper.

Total solids. Total solids content was determined by drying a tared sample in a metal dish, provided with a cover, in an oven at 95° C. till most of the free water was removed. Drying was then continued for a further 20 hours at 70° C. and a vacuum of 28 inches of mercury.

Water insoluble solids. This was determined by the A.O.A.C. *(1)* procedure. Prepared Whatman No. 54 paper was used for the filtration.

Soluble solids. This was determined with a Zeiss opton refractometer and readings were corrected to 20° C.

Comparative serum viscosity. This was determined in a falling ball viscosimeter after accurate dilution of the original serum to 5° B. The same ball was used throughout and the temperature held at 20.0 ± 0.1° C. The time necessary for the ball to fall through 5 cm. was recorded.

Calcium pectate. Calcium pectate was determined according to the method described by Kertesz *(4)* except that after precipitation of calcium pectate the precipitate was filtered on a washed, dried and weighed Whatman No. 54 paper. The precipitate was thoroughly washed with hot water, followed by three washings with 95% ethanol and three with ether. The paper was then dried overnight at 95–100° C. before cooling and weighing.

Methoxyl content. This was determined by saponification with alkali. An aliquot of 70 g. of serum was weighed into a 250 ml. beaker and titrated to pH 7.5 by adding 0.4 N sodium hydroxide. The end point was determined with a pH

meter while the aliquot was stirred with a magnetic stirrer. The neutralized solution after a further 20 ml. of 0.4 N NaOH was added, was left for exactly 1 hour at ± 25° C. The excess alkali was then titrated with 0.2 N HCl to pH 7.5. A blank containing no tomato serum was run at the same time.

Juice settling. Juice settling was measured by adjusting the original juice before concentration to 5° B. with distilled water and then filling to the mark in identical 100 ml. measuring cylinders. The volume of serum formed on top was taken as an indication of the degree of settling.

RESULTS AND DISCUSSION

The change in consistency as measured with the Bostwick consistometer during concentration of San Marzano and Pearson juice with the various treatments is illustrated in Figure 1. These results were obtained during the 1957 season and were essentially the same as those obtained during the previous season. Only one year's results are therefore given.

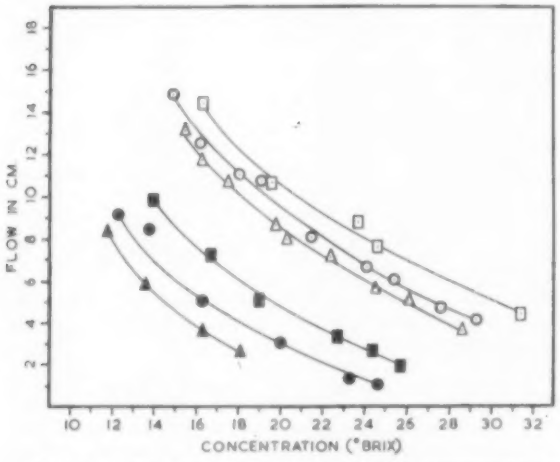

Figure 1. The change in consistency during cocnentration of tomato juice.

Legend:

- ⊡ Pearson, no delay between breaking and preheating, 0.02 inch screen.
- ⊙ Pearson, no delay between breaking and preheating, 0.04 inch screen.
- △ Pearson, 3 hours delay between breaking and preheating, 0.04 inch screen.
- ■ San Marzano, no delay between breaking and preheating, 0.02 inch screen.
- ● San Marzano, no delay between breaking and preheating, 0.04 inch screen.
- ▲ San Marzano, 3 hours delay between breaking and preheating, 0.04 inch screen.

In all cases where the tomatoes were rapidly heated after breaking the paste obtained with an 0.02 inch screen in the finisher had a lower consistency than when an 0.04 inch screen was used. This substantiates the observations of Hand *et al. (3)* on the effect of screen size on the gross viscosity of tomato juice.

A more important finding however, was the higher consistency always obtained in those cases where a delay of three hours between breaking and preheating was allowed as compared with those immediately preheated after breaking. This observation is inconsistent with the results of several other workers *(5, 6)* who obtained poorer consistencies or gross viscosities after a delay between breaking and inactivation of enzymes by heat. In most of this work however finishing was done at a low temperature which could give a lower amount and a different type of insoluble material in the final juice, as well as a lower pectin content when compared to juice which was rapidly heated after breaking and finished at elevated temperatures. In fact, Hand *et al. (3)* showed that holding at 70° F. and 130° F. between chopping or breaking and preheating had no marked effect on gross and serum viscosity of tomato juice. They suggested however that less disruption in the chopper might have influenced their results. This seems to be a plausible explanation because their serum viscosities as influenced by pectin content did not show much difference from samples where no delay occurred before preheating and finishing.

The analytical results obtained with the various pastes during the 1957 season are compiled in Table 1. The results clearly demonstrate that a delay of 3 hours at 70–80° F. between breaking and preheating gave a very marked decrease in the calcium pectate and methoxyl contents as well as in the comparative serum viscosity. In spite of this the consistency was higher.

From past experience it is well known that pectic substances play an important role in determining consistency and in Table 2 are given the results obtained when diluted San Marzano paste was treated with 0.2% Pectinol A for 24 hours at 40° C. It is evident that Pectinol treatment resulted in a poorer consistency, lower comparative viscosity and a higher degree of settling.

The higher consistency of the pastes prepared from juice delayed for three hours before preheating, could not be due to the amount of water insoluble substances present because it was very much the same with the two treatments. From the results of this study and the recent work of Hand *et al. (3)* and Whittenberger and Nutting *(10)* it would seem reasonable to assume that a delay of three hours between breaking and preheating caused the insoluble particles in the final paste to be structurally different from that of a paste in which the tomatoes were immediately heated after breaking. This structural difference in the delayed paste resulted in a higher consistency in spite of a lower pectin content.

SUMMARY

San Marzano and Pearson tomatoes were manufactured into paste by using two different screen sizes. Some fruit samples were immediately heated to 180° F. after breaking, while others were delayed for three hours at 70–80° F. between breaking and preheating. A larger screen size in the finisher gave a paste with a higher consistency. Delay between breaking and preheating gave a higher consistency at the same total soluble solids level than samples which were

TABLE 1

Influence of treatment on composition, consistency, settling and comparative serum viscosity of tomato juice and paste

Variety	Delay between breaking and preheating to 180° F. (hours)	Screen size on finisher (inches)	Bostwick consistometer reading at 17° Brix (cm.) after 1 minute	Comparative viscosity of serum (seconds)	Settling of 5° B juice during 24 hours at room temp. (ml. clear juice on top)	Water ins. solids % (on 5% total solids basis)	Calcium pectate % (on 5% total solids basis)	Methoxyl content % (on 5% total solids basis)
Pearson	0	.04	12.1	96	7.6	0.39	0.096	.0138
Pearson	0	.02	13.5	92	11.0	0.35	0.100	.0124
Pearson	3	.04	11.1	79	9.2	0.38	0.043	.0100
San Marzano	0	.04	4.6	116	6.9	0.48	0.153	.0133
San Marzano	0	.02	6.8	101	9.8	0.43	0.131	.0143
San Marzano	3	.04	3.2	95	5.0	0.52	0.078	.0113

TABLE 2

Effect of Pectinol A on consistency, comparative serum viscosity and settling of tomato paste

Sample	% Pectinol used during treatment at 40° C. for 24 hours	Bostwick consistometer readings at 15.2± 0.2° B (cm.) after 15 seconds	Comparative viscosity of serum (seconds)	Settling of diluted 5° B paste during 24 hours at room temp. (ml. clear juice on top)
San Marzano. Delayed 3 hours between breaking and preheating. .04 in. screen in finisher	0	7.0	104.6	8.9
	0.2	12.6	66.2	13.6
San Marzano. No delay between breaking and preheating. .04 in. screen in finisher	0	5.5	93.0	4.0
	0.2	9.1	63.0	10.7

immediately preheated after breaking, even though the calcium pectate, methoxyl content and comparative serum viscosities were lower.

As the water insoluble solids content was rather similar for the two treatments, it is suggested that the difference in consistency was due to a qualitative difference in the insoluble substances.

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Factors Affecting Loss of Nitrogen and Fermenting Power of Rehydrated Active Dry Yeast*

R. K. Sant and W. H. Peterson

Department of Biochemistry, University of Wisconsin, Madison, Wisconsin

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COMMERCIAL ACTIVE DRY YEAST (ADY) is very sensitive to the temperature of the water in which it is rehydrated. If the yeast is resuspended in water at about 40° C., it gives a well-raised bread but if the temperature of the water is in the neighborhood of 5° C. the volume of the loaf is reduced and the bread is unacceptable. Manufacturers of ADY are well-aware of this situation and instruct users to resuspend the yeast in warm water before making the dough. Users may be careless about following directions and hence may unjustly blame the yeast for the poor results that follow. Peppler and Rudert *(14)* were the first to call attention to the effect of cold water on ADY. They showed both by staining methods and plate counts that about one-third of the yeast cells were inactivated by rehydration at 5–10° C. and also reported that the baking strength of the yeast was markedly reduced by rehydration at low temperature.

A later paper by Herrera *et al. (5)* reported that about 25% of the yeast solids were extracted at 4.5° C. and nearly all of the soluble nitrogen of the cell was lost at this temperature. The leached nitrogen consisted mainly of free amino acids, of which glutamic, alanine, and valine were the most abundant. Large losses of trehalose, phosphorus and niacin and smaller losses of thiamine and riboflavin also occurred. The same authors found that carbon dioxide production by yeasts reconstituted at the low temperature was less than one-fourth that given by yeasts treated with water at higher temperature.

Dried bacterial cells as well as ADY are greatly affected by the temperature of rehydration. Hiscox *(7)* and Higginbottom *(6)* found quite regularly 5 to 10 times as many bacteria in spray dried milk powders when the samples were rehydrated at 50° C. as at room temperature, 18 to 22° C. In one series of platings, the counts increased six-fold with higher rehydration temperatures beginning at 20° C. and going up by 5° C. intervals to 50° C. Speck and Myers *(15)* reported that the viability of spray dried milk cultures of *Lactobacillus bulgaricus* was increased as much as 5 times by rehydration at 50° C. instead of at 20 to 25° C.

All of these authors noted that it was the initial temperature of the liquid that determined the number of viable cells. Raising the temperature to 50° C. after suspension at 21 to 25° C. did not increase the number of viable cells. It has been suggested that the higher temperature has a "shock" effect on the cells just as spores are stimulated to growth by being subjected to high temperatures for a short time. An alternative explanation would be that the cells reconstituted at room temperature lost so much of the cell constituents that they were unable to grow. Some data on this aspect of the problem have been obtained for *Serratia marcescens* by Johnson and coworkers *(8)*. From 20 to 30% more organic nitrogen (*e.g.* amino acids and peptides) was lost on rehydration at 0° C. than at 25° C. Rehydration at 0° C. killed practically all of the cells and at 7° C. about one-third as many survived as at 24° C. Wasserman and Hopkins *(16)* recently reported that 95% of the cells of *S. marcescens* that grew when rehydrated at 30° C. failed to do so when reconstituted in water at 5° C.

The present paper gives some data on attempts to prevent the loss of soluble nitrogen from ADY at low temperatures of rehydration and to correlate the losses with the physiological activity of the yeast cells.

EXPERIMENTAL

In all the experiments the active dry yeast (ADY) was that kindly supplied by Red Star Yeast and Products Company, Milwaukee.

Treatment of yeast. One-half or one gram of ADY was weighed and kept in a beaker which was placed in an ice bath at 4.5° C. or in a water bath at 37° C. depending upon the temperatures chosen for rehydration. Ten milliliters of distilled water (with or without various additives) having the corresponding temperature was added to the yeast and the whole stirred gently for 15 minutes. The suspension was then used for making plate counts and for determining carbon dioxide production. After centrifuging, the supernatant was used for nitrogen determination.

The process of taking up moisture by ADY from water vapor in the surrounding air will be referred to as rehumidification, and rehydration is used to denote the reconstitution of the yeast cells in water. One-half gram of ADY was weighed in special containers with nylon screen bottoms, obtained from the Red Star Yeast and Products Co. The sample was placed in a desiccator containing a petri dish partly filled with water and held for various intervals of time at the desired temperature. It was then weighed again to determine the moisture taken up. Immediately after the final weighing the yeast was rehydrated in water at the same temperature as was used for rehumidification and was then treated in the usual way for analysis. Experiments were run at 3 temperatures, 4.5° C., 26° C. and 37° C.

Methods of analysis. Nitrogen was determined on the supernatant by the micro-Kjeldahl method *(9)* and the quantity of nitrogen extracted from the cells was taken as a measure of the loss of cell constituents. CO_2 production was taken as an index of the activity of the cells. One-half gram of ADY was rehydrated in 10 ml. water at 4.5° C., 26° C., or 37° C. for 15 minutes, diluted to 250 ml. with distilled water and an aliquot used for carbon dioxide production in a Warburg respirometer. One-half milliliter of the suspension, equivalent to about 1 mg. dry yeast, was added to a Warburg flask containing 2.5 ml. of the medium used by Oyaas *et al. (13)*. Anaerobic conditions were obtained by means of a nitrogen wash.

* This investigation was supported in part by a research grant from Red Star Yeast and Products Company, Milwaukee, Wisconsin.

For plate counts 1 ml. suspension of yeast was added to 99 ml. saline (0.85% NaCl) and agitated for 10–15 seconds in a Waring blender. All subsequent dilutions were made in saline. Plates were poured with nutrient agar fortified with 1% glucose, incubated at 30° C. for 48–60 hours and then counted.

RESULTS AND DISCUSSION

Loss of nitrogen at 4.5° C. ranged around 10 mg. per g. of ADY, while at 37° C. the quantity was about 5 mg. Based on the total nitrogen of the yeast, the loss was 15% at 4.5 ° C. and about 7% at 37° C. Nitrogen losses at the two temperatures are in good agreement with the figures reported by Herrera and coworkers *(5)*. Soluble nitrogen in this sample, obtained by extraction with boiling water, was 16.4% of the total nitrogen. The loss of nitrogen at 4.5° C. represents most of this soluble nitrogen which may be regarded as the working capital of the cell.

Effect of various treatments on leaching. Since rehydration of ADY at 4.5° C. resulted in the loss of soluble compounds and reduced activity, a method to prevent this effect was sought. Results of these attempts are given in Table 1.

In the first experiments pretreatment of the cells with very small quantities of melting snow or water at 4.5° C. for varying lengths of time were tried. The quantity of snow or water was less than that contained in a fully rehydrated cell and it was hoped, would leave no excess of water outside of the cell. The loss of nitrogen from the pretreated cells was about 10 mg. per g. regardless of whether the subsequent rehydration was done at 4.5° C. or at 37° C. Conversely, ADY pretreated with the same small quantities of water at 43° C. and then rehydrated at 4.5° C. suffered no more loss (about 5 mg. per g.) than if rehydrated at 43° C. It is the temperature of the initial exposure that determines the loss and not subsequent temperatures or treatments. In the next two runs a small quantity of water containing adenosinetriphosphate (ATP), oxidized diphosphopyridine nucleotide (DPN), and glucose, were added to ADY at 4.5° C. Keeping the sample at 4.5° C. for a long time or subsequently holding it further at 24° C. with the object of increasing the metabolism did not inhibit the loss of nitrogen. It is possible that these substances were not able to enter the cell and exert an effect before the loss occurred.

In another experiment the concentration gradient across the cell wall was increased by adding 1 ml. of concentrated ADY extract to 1 g. of ADY at 4.5° C. and holding for 30 minutes at this temperature and then rehydrating in 9 ml. of water at 4.5° C. in the usual way. The extract was obtained by rehydrating 200 g. ADY in 2 liters of water at 4.5° C., removing the cells by centrifugation and concentrating the supernatant under reduced pressure to 200 ml. One ml. of this concentrated extract was therefore equivalent to leachings from 1 g. of ADY. This extract was thus 10 times as concentrated as that obtained in the usual test samples and about three times that in the rehydrated cell. Data in Table 1 show no decrease in the loss of nitrogen. Therefore raising the concentration of nutrients outside the cell to a level three times

TABLE 1

Effect of various treatments of ADY on loss of nitrogen

Series	Rehydration conditions				Nitrogen lost
	Initial		Secondary		
	0° C.	*min.*	*0° C.*	*min.*	*mg./g.*
I. Pretreatment/g. ADY[1]					
0.2 g. snow	4.5	30	none	none	10.5
0.2 g. snow	4.5	30	24	90	12.0
0.5 ml. water	4.5	1440	none	none	10.4
0.5 ml. water	4.5	30	43	30	12.2[2]
0.5 ml. water	37.0	30	4.5	60	5.9
0.6 ml. water[3]	4.5	30	none	none	10.9
0.6 ml. water[3]	4.5	30	24	90	11.0
1.0 ml.conc. ADY extract	4.5	30	none	none	10.7
1 g. 'Certo' pectin	4.5	30	none	none	12.2
II. Rehydration/g. ADY[4]					
10 ml. 25% glucose	4.5	15	none	none	10.6
10 ml.0.01 mM $CaCl_2$	4.5	15	none	none	10.8
10 ml. 0.1 mM $CaCl_2$	4.5	15	none	none	10.9
10 ml. 1 mM $CaCl_2$	4.5	15	none	none	11.9

[1] In series I the cells were pretreated as mentioned in column 1 before rehydration.
[2] Rehydrated at 37° C.
[3] Contained 9.8 mg. ATP, 0.52 mg. DPN and 100 mg. glucose.
[4] In series II there was no pretreatment and the rehydrating solution was that given in column 1.

that in the rehydrated cell was not enough to produce an effective concentration gradient across the cell wall. Similar results were also obtained when ADY was rehydrated at 45° C. in 25% glucose solution.

Improvements in the drying characteristics and activity of ADY have been reported *(1)* when fruit pectin was added to the yeast before drying. The effect of pectin if any when added to ADY before rehydration was determined in one of the experiments. The pectin was a commercial product, 'Certo', obtained from apple juice. From the data it can be seen that the loss of nitrogen was not prevented; on the contrary a slight increase above the normal loss was noticed. Apparently, to have an effect, pectin must be dried on to the yeast.

Calcium is a prominent component of the cell membrane *(2)*. Davson and Danielli *(4)* have shown that calcium chloride increases the resistance of the cell membrane to penetration and conceivably might prevent escape of cell constituents. In order to observe the effect of calcium ions on the loss of nitrogen, ADY was rehydrated in varying strengths of calcium chloride solutions at 4.5° C. No reduction in the nitrogen loss was observed.

Vapor rehumidification and rehydration. Since ADY is made by evaporating the water from the cell, it seemed logical to assume that the cell could be restored to its original state by reversing the process, *i.e.*, by vapor rehumidification. Cells were rehumidified in an atmosphere saturated with water vapor at 3 different temperatures. They then were suspended in water at the same temperature that was used for humidification and the loss of nitrogen determined. Results of this experiment are summarized in Table 2. Rehumidification and rehydration at 4.5° C. produced no significant change in the loss of nitrogen, until the moisture content of ADY reached 20%. Then the loss was only 6.0 mg. per g.—about the same as that of yeast with 8% moisture when this was rehydrated at 37° C. When the moisture content reached 25%, the loss of nitrogen was reduced to 3.7 mg. per g. which is

TABLE 2

Effect of vapor rehumidification on loss of nitrogen in subsequent rehydration

Time	Results at 4.5° C.		Results at 26° C.			Results at 37° C.		
	Moisture	N. lost	Time	Moisture	N. lost	Time	Moisture	N. lost
hr.	%	*mg./g.*	*hr.*	%	*mg./g.*	*hr.*	%	*mg./g.*
0	8	10.8	0	8	8.0	0	8	5.8
2	17	10.3	1	15	3.0	1	14	3.0
3	20	6.0	2	20	2.2	2	20	3.5
6	25	3.7	3	23	2.2	3	27	3.6

about the same as that incurred at 37° C. with yeast of the same moistur content. In other words the effect of low temperature rehydration on the loss of nitrogen was eliminated.

Loss of nitrogen from ADY containing 8% moisture, on rehydration at 26° C. was 8.0 mg. per g.—a value about half way between the losses at 4.5 and 37° C. When the moisture content of the yeast was increased to 15%, nitrogen loss was reduced to 3.0 mg. per g., while at a moisture content of 20% the loss was only 2.2 mg. per g. ADY with a still higher moisture content did not show any further reduction in loss of nitrogen.

Results of rehumidification and rehydration at 37° C., show a considerable reduction in the loss of nitrogen when the moisture content reached 14% but no further reduction at higher moisture levels.

Some additional experiments were made with cells rehumidified at both 4.5° C. and 37° C. to the apparently critical moisture content of 20–25% and then rehydrated at both of these temperatures. Such cells lost about the same small amount of nitrogen at either 4.5° C. or 37° C. When the cells regained a moisture content of 20–25%, they were apparently completely reorganized and were then independent of the former low temperature effects.

Effect of vapor rehumidification on activity of ADY. As a measure of the physiological state of the rehydrated cells, CO_2-production and plate counts seemed the most logical criteria. Yeast samples were rehumidified as before at 3 different temperatures and then dispersed in water at the same temperature as was used for rehumidification. Suitable aliquots were taken for CO_2-production in the Warburg respirometer and plated for cell counts. The data are given in Table 3.

Results at 37° C. may be taken as a reference point for ascertaining the number of potentially viable cells and CO_2-producing ability. Rehumidification had very little effect on either index. The cell counts are in the region of those obtained per g. of dry solids for undried cells.

The data for the samples treated at 26° C. show a moderate increase in CO_2-production as a result of rehumidification but the cell counts do not reflect any consistent change. The number of viable cells and CO_2-production at the 8% moisture level seem unexpectedly high in view of the rather large loss of nitrogen from the cells on rehydration (Table 2). These cells lost 8 mg. of N per g. and yet practically all of the cells remained viable. This suggests that the damage done at 4.5° C. is due to something more than loss of soluble constituents—perhaps the enzymes of the cell are damaged.

Perhaps the most important data in Table 3 are those pertaining to the 4.5° C. samples. Cell counts and CO_2-production were very low. Only 5% of the cells survived rehydration when their moisture content was 8% but 40% remained viable when the cell moisture was increased to 17% and practically all the cells withstood low temperature rehydration when the cells contained 22–25% moisture.

CO_2-production by rehydrated cells increased markedly as the initial moisture content went up—more than 4-fold in going from 8 to 17% moisture and about 8 times on reaching 22% moisture. At this level, the cells gave nearly the same production of CO_2 as cells resuspended at 37° C. These data point to the importance of water in the organization of the yeast cell. A minimal amount of it is necessary for the cell to be independent of external temperature conditions.

Proposed interpretations. The approximately 8% moisture content of commercial ADY appears to be a practical compromise between moisture levels that increase activity but reduce stability and those that increase stability but reduce activity. Crane *et al. (3)* and Morse and Feller *(11)* report that yeasts with more than 8% moisture had good activity although the shelf life of the yeasts was reduced. Olhaver *(12)* found that the drier the yeast the greater was its stability but the poorer was its leavening power. However if these yeasts were first rehumidified they regained their activity. In Mitchell and Enright's *(10)* experiments, yeast of 7.8% moisture became unacceptable for bread making after storage at 115° F. for 1 week. Samples containing 5.5, 4.0 and 2.2% moisture withstood this temperature for 6, 12 and 24 weeks respectively if the samples were rehumidified to the original moisture content before being used. However yeasts dried to 0% moisture made poor bread even if rehumidified before use. No

TABLE 3

Effect of vapor rehumidification on activity of ADY

Moisture content	Results at 4.5° C.		Results at 26° C.		Results at 37° C.	
	CO_2 prod. in 120 min.	Plate count	CO_2 prod. in 120 min.	Plate count	CO_2 prod. in 120 min.	Plate count
%	*ml./g.*	*per g.*[1]	*ml./g.*	*per g.*[1]	*ml./g.*	*per g.*[1]
8	92	0.48	690	9.9	734	10.0
17	401	4.0	794	10.4	765	10.4[2]
20	716[3]	9.2[3]	787	8.8	776[2]	11.0
25	693	11.0	755	9.8	776	10.0

[1] 10^8 times omitted.
[2] Moisture content was 14%.
[3] Moisture content was 22%.

data are available on the loss of cell constituents in these experiments.

The question naturally arises as to why low temperature rehydration destroys the viability and activity of the yeast cells. It seems improbable that the enzymes of the cell are damaged. These are apparently retained within the cell and low temperatures are generally regarded as a means of preventing loss of enzyme activity. It is likewise improbable that water at low temperature does any damage to the cell wall. The most evident and logical interpretation then is that the loss of the cell constituents makes it impossible for the enzymes within the cell to operate. These constituents are probably the substrates with which the enzymes act. If they are absent, the enzymes cannot function just as an automobile engine cannot operate if there is no fuel in the gas tank. From previous work *(5)*, it appears that about 25% of the solids of the cell are extracted at 4.5° C. and only 6 to 10% are removed at 43° C. While there is some variation in the composition of the extracts at the two temperatures, the soluble non-protein nitrogen extracted may be taken as a fair index of the loss of total solids. At 26° C. about 80% of this nitrogen was lost and yet practically all of the cells remained viable and had excellent CO_2-producing ability. Apparently it is the loss of the last 20% of the soluble constituents that does the damage. Whether what remains in the cell at 26° C. is of different composition than what is extracted can only be decided by additional analytical data. It is not yet possible to say which, if any, of the extracted compounds is specifically responsible for the loss of viability and activity. If a guess is permissible, probably there are a number of such compounds in the extract. If we knew more about the physical state of the water in the cell and what role it plays in the organization of the cell, the picture might be clearer. It appears that a moisture content of about 25% is required for withstanding rehydration at 4.5° C., 15% is sufficient for protection at 26° C., and 8% is adequate for retention of viability and activity at 37° C.

SUMMARY

Active dry yeast (ADY) lost about twice as much nitrogen when reconstituted at 4.5° C. as at 37° C. The excessive loss at 4.5° C. could not be prevented by reconstituting the cells in concentrated extracts of the cells or in minimal quantities of water containing coenzymes, pectin, calcium chloride or high levels of glucose.

Vapor rehumidification of ADY decreased the loss of nitrogen on subsequent reconstitution. A moisture content of about 25% appears to be the critical level for protection against cell damage by cold water.

Suspension of ADY in water at 4.5° C. killed about 95% of the cells as judged by plate counts and reduced their CO_2-producing ability about 90% below that of samples rehydrated at 37° C. If the moisture content of ADY is raised to 25% by vapor rehumidification, suspension in water at 4.5° C. has no ill effects on the number of viable cells or their CO_2-production. Tests at 26° C. gave results more nearly like those at 37° C. Full activity was reached at a moisture content of about 17%.

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Pineapple Juice Powder

G. K. Notter, D. H. Taylor and J. E. Brekke

Western Regional Research Laboratory,* Albany, California

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POWDERS PREPARED from fruit juices or fruit juice concentrates have been described in several publications in the past few years *(1, 2, 8, 10, 11, 14)*. Orange and grapefruit juice powders are produced commercially. This is a report on the preparation of a powder from pineapple-juice concentrate by means of a vacuum shelf dryer.

MATERIALS

The material from which the powder was made is a commercially available, hot-pack, 47° Brix pineapple-juice concentrate. After drying, the screened powder was packed in hermetically sealed cans (95 g. per can) with a small packet containing 10 g. of calcium oxide which acts as an "in-package desiccant" (IPD). Packaging was done in an atmosphere below 10% relative humidity and where indicated, a vacuum of 28 inches Hg was used.

ANALYTICAL METHODS

Moisture content of powders was determined as weight lost upon drying in a vacuum oven for 30 hours at 60° C. and 0.6 mm. Hg pressure. This method gave results in agreement with the McComb and Wright modification of the Karl Fischer method *(6)*. Suspended pulp was measured on material adjusted to contain 15% soluble solids; this was centrifuged 3 minutes at 1270 r.p.m. at a diameter of 16 inches *(15)*. Ascorbic acid analyses of feed and powder were carried out by the Loeffler and Ponting method *(5)*. Reductones were assayed by the method of Wokes *et al. (18)*. Sulfur dioxide was determined by a modification of the Monier-Williams method (7). Soluble solids were determined refractometrically. Moisture equilibria were determined with the apparatus of Legault, Makower, and Talburt *(4)*. This method was applied only in the moisture range where caking does not occur at the temperature of the determination. The method of Wink *(17)* using saturated salt solutions was applied over the range 11 to 23% relative humidity; sulfuric acid solutions *(9)* were used for relative humidities below 11%. Esters were determined by a modification of the method of A. R. Thompson *(13)* in which the esters are separated by distillation rather than alcohol extraction. A sample (100 to 200 g.) is made up to 400 ml. with distilled water; 35 ml. of distillate is collected in a receiver containing 50 ml. of water and cooled by an ice bath. The method gives 94% ± 3 recovery of volatile esters.

Bulk density was obtained by weighing powder into a graduated cylinder and noting the volume before and after the cylinder was rapped 50 times on a wooden surface.

Sieve analysis was performed by shaking a sample through a graduated series of sieves for 5 minutes on a mechanical shaker.

Color measurements were made on the extract from a 5% aqueous solution of the powder; the extract is clarified by filtration with analytical grade Celite through a medium-porosity fritted-glass filter. A Klett colorimeter with a 4-cm. cuvette and equipped with a 400 to 465 mμ filter was used for determining color; results are expressed as Klett units.

Flavor evaluations were carried out by two types of panels. Triangle and paired taste comparisons were made by a panel of judges trained to detect differences in fruit and fruit juices. Tasting was done in panel booths illuminated with dim colored lights to prevent sample detection by appearance. In addition to formal panels, 6 judges evaluated and commented on quality difference between paired samples. Remarks concerning coded samples were recorded by each judge, and discussion between judges was not allowed until after all records were completed. All samples were reconstituted to 15.0° Brix for tasting. Where triangle taste comparisons were made, probability values were taken from the *Tables of the Binomial Probability Distribution*, U. S. National Bureau of Standards *(16)*.

RESULTS

Time and temperature limits. Pineapple-juice concentrate (47° Brix) was spread on trays at 0.5 lb. per sq. ft. and dried at 2 mm. Hg pressure under various conditions of time and temperature. A powder below 3% moisture was desired. It was found that satisfactory yield and quality were obtained if drying was done at 150° F. for 2¾ hours. For 140 minutes of this time the product temperature was below 150° F., as can be seen in Table 1.

The loaded trays were placed on shelves and about 5 minutes were required to lower the pressure to 2 mm. Hg. Then heat was applied through the hollow shelves by circulating hot water. After drying, the product was cooled by circulating cold water in the shelves before the drier was opened.

The rate of drying at 150° F. shelf temperature and 2 mm. Hg pressure was investigated. In Figure 1 the moisture content of this product plotted with drying time shows that in 3 hours the moisture content was reduced to about 2%. The material dried 2¾ hours contained only 2.5% moisture and was easily removed from the trays. Damp spots appeared in the trays dried only 2¼ and 2½ hours; the damp spots indicated uneven drying and made recovery of the product impractical.

The powder (2.5% moisture) was easily removed from drying surfaces and readily passed a 6-mesh screen. The bulk density of the powder was about 0.30 g. per ml. when compacted. About 35% of the powder was retained on 20-mesh screen, 35% on 40-mesh, and of the remainder, one-half was retained on 60-mesh.

Pressure. Operating pressure of 4 mm. Hg was

TABLE 1

Drying conditions: effect on moisture content and yield

Shelf temp.	Minutes product to attain shelf temp.	Minutes product at shelf temp.	Moisture	Yield
° F.			%	%
100	360	600	3.5	90
140	205	35	1.8	90
140	200	100	1.7	90
150	140	40	2.1	90
150	145	95	1.2	90
160	120	0	2.2	65
160	120	30	1.5	65

(47° Brix concentrate 0.5 lb./ft.², 2 mm. Hg pressure)

* Western Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture. Presented at IFT meeting Pittsburgh, Pa., June 1956.

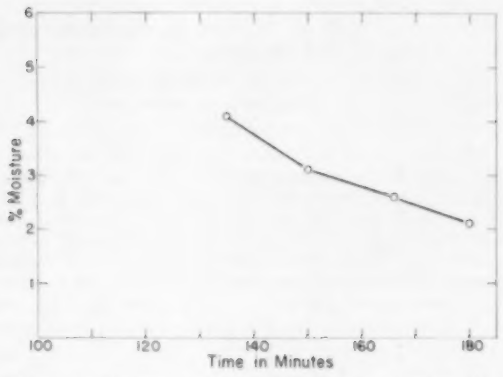

Figure 1. Moisture content of powder vs. drying time of concentrate in vacuum shelf dryer at 150° F.

found to be unsatisfactory for drying 47° Brix concentrate. However, a 63° Brix concentrate was dried satisfactorily at 6 mm. Hg pressure. Data in Table 2 show relationships between Brix of starting material, operating pressure, yield, and moisture content of product. Optimum tray loading for concentrates of 47° to 63° Brix was 0.5 lb. per square foot.

Pulp. Pulp increases the bulk density of the powder. Samples were prepared from 47° Brix concentrate containing 1%, 16%, and 32% pulp *(15)*. These samples gave dried products of open porous structure (puff) of depths of 2 inches, ¾ inch, and ¼ inch on the tray; the bulk densities of 40-mesh powders were 0.34 g./cc., 0.53 g./cc., and 0.70 g./cc. The moisture contents of these powders were 2%, 2.5%, and 3%, respectively.

Flavors and esters. Pineapple juices prepared from powder and from concentrate were compared by a triangle taste panel. The samples were distinguished correctly 19 times in 41 judgments ($P = 0.052$). Five judges preferred the sample prepared from powder and 13 preferred that prepared from the concentrate.

Volatile esters appear to contribute to the flavor of pineapple juice and their presence in the powder has some effect on the flavor of reconstituted juice. A commercial 47° Brix concentrate containing 75 p.p.m. esters, moisture-free basis (MFB), yielded a powder containing 17 p.p.m. (MFB). Loss of esters was shown to take place at an early stage in drying: samples taken at 51°, 55°, and 60° Brix during the drying cycle (starting with a cold shelf) all had 17 p.p.m. esters (MFB) as did the finished powder. When the starting shelf temperatures were 140° F. and 160° F., the ester contents were 12 and 8 (MFB), respectively, in samples that had reached 60° Brix.

Powder containing less than 1 p.p.m. ester (MFB) was prepared from concentrate from which volatile esters had been removed. Samples of this powder were reconstituted and fortified with various amounts of a 60-fold commercial pineapple essence. Using the paired taste test with 30 judgments, a two-fold difference in ester content was uniformly detectable ($P = 0.05$) over the entire range of 2.7 to 14 p.p.m. esters. This indicates that maximum retention of volatile esters is desirable.

Incorporation of air into the concentrate prior to drying diminishes the ester content in the dried product. A 47° Brix concentrate (75 p.p.m. ester, MFB) beaten 5 minutes with a mechanical mixer showed a 25% increase in volume. This material was dried at 100° F. for 16 hours at 2 mm. Hg, giving a powder containing no esters and 3.0% moisture. A powder prepared from identical concentrate that had no air beaten into it contained 11.6 p.p.m. esters and 3.7% moisture.

Volatile ester material was added to 63° Brix concentrate and to 47° Brix concentrate prepared by dilution of the former. Both samples were adjusted to contain 140 p.p.m. (MFB) volatile esters. They were dried for 16 hours at 100° F., 2 mm. Hg pressure. The powders contained 5.5 p.p.m. and 1.8 p.p.m. volatile esters, respectively; moisture contents were 2.5% and 3.0%. The loss of esters in this experiment was greater than that encountered upon drying of commercial 47° Brix concentrate; samples of commercial concentrate containing 75 p.p.m. (MFB) volatile esters yielded powders with 17 p.p.m. volatile esters. The ester content of the powder was about the same whether long (16 hours, 100° F., 2 mm.) or short cycle (2¾ hours, 150° F., 2 mm.) drying was used and was independent of the final moisture content. A taste panel could not differentiate (10 correct judgments out of a total of 31) between the short and long cycle powders. A powder containing 3.7% moisture and 38 p.p.m. ester (MFB) was stored 8 months at 77° F. with an in-package desiccant. At completion of this storage the powder contained 1% moisture and 38 p.p.m. volatile ester (MFB). A powder containing no sulfite and less than 1 p.p.m. esters (MFB) was stored at 100° F. for 30 days; scorched flavor developed, indicating that off-flavors were not due to deterioration of retained esters.

Moisture equilibria. Powders at different moisture levels are in equilibrium with atmospheres containing different amounts of water vapor. These equilibrium points were measured by Dubrovin gauges *(4)* and with sulfuric acid solutions *(17)*. The data are plotted in Figure 2 with both vapor pressure of water (mm. Hg) and equivalent relative humidity noted on the abscissa. It is clear that powder containing 2.5% moisture should be handled (i.e., detrayed and packaged) in a room of 10% relative humidity or below.

TABLE 2

Product yield and moisture as related to degrees Brix of starting material and operating pressure

Pressure mm. Hg.	Degrees Brix	Yield	Moisture
		%	%
2	41	78	2.5
	47	93	2.5
	51	94	2.4
	63	92	2.4
4	47	70	3.0
	63	94	2.0
6	47	64	3.4
	63	97	2.5

(150° F. shelf temp.; 2 hours, 45 minutes; 0.5 lb./ft.²)

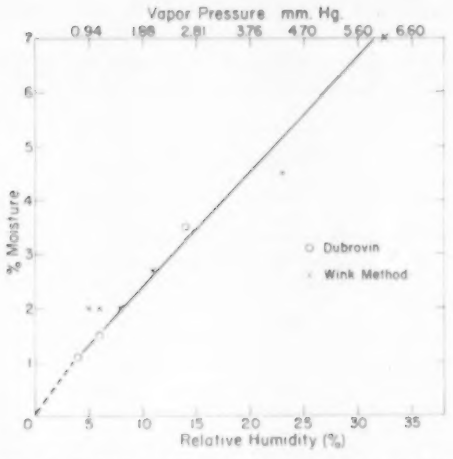

Figure 2. Moisture-relative humidity equilibria of pineapple juice powders at 70° F.

In-package desiccation (IPD). The use of in-package desiccation (IPD) and controlled storage temperatures immediately after packaging have been shown to prevent caking of powders and enhance their stability chiefly as a result of further reduction in moisture content *(10, 11, 12)*. The caking of pineapple juice powder, in the absence of IPD, was studied by storing powders of different moisture levels in glass jars at several temperatures. A sample was considered caked if a sharp rap on a wooden surface failed to loosen the powder in the jar. Caking increased with moisture content and storage temperature (Table 3).

During the period of controlled storage immediately after packaging with IPD—the so-called "conditioning" period—the moisture content of the powder decreases at a rate dependent on temperature of storage. The objective in this work was to bring the moisture content to a level at which the powder would not cake in storage. The rate of moisture loss by pineapple juice powder (Figure 3) is less than rates reported for orange juice powder *(8)* and lemonade powder *(10)*. Pineapple juice powder was held 220 days at 77° F. with IPD to reduce the moisture content from 2.6 to 1.0%. At 90° F. this powder reached 1% moisture in 35 days. However, powder stored at 90° F. showed some deterioration of flavor, which suggested that controlled storage, or conditioning, should be started at least at a temperature below 90° F.

TABLE 3

Days to cake (no IPD)

Temperature, ° F.	Moisture, %						
	1.8	2.1	2.3	2.7	3.0	3.2	3.7
	days						
100	2	2	2	2	2	2	2
90	4	4	2	2	2	2	2
85	14	14	9	6	4	4	2
80	21	21	14	6	4	4	2
77	63	56	42	14	9	7	2

A rapid or "cascade" conditioning was devised in which the storage temperature was gradually increased as moisture of the powder decreased. The data in Table 4 show the progress of this rapid conditioning method. About 65 days of storage was sufficient to achieve a moisture level of 1%. Samples containing 2.5% moisture packaged with IPD could be placed in 90° F. storage directly from the drier without caking.

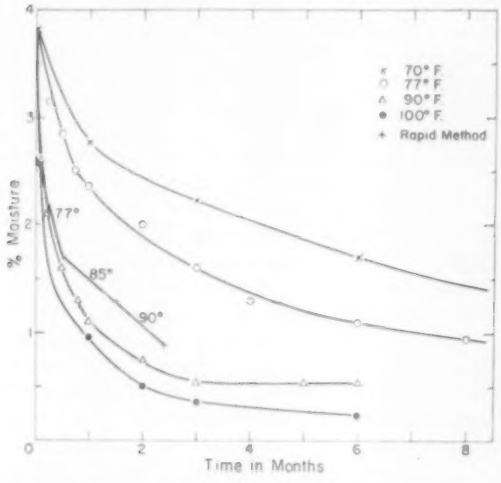

Figure 3. Effect of time and temperature of storage (with IPD) on moisture content of pineapple juice powder.

When it is known that the package will be held at temperatures below 90° F., it appears unnecessary to add to storage by conditioning. Temperatures of 100° F. invariably introduce the problem of caking and obvious flavor damage.

If powder storage is to be such that conditiioning is desirable, the cascade method would be preferable to holding at a constant low temperature because less holding time is necessary. The triangle taste panel

TABLE 4

Moisture changes during rapid ("Cascade") conditioning

Temperature	Days	Moisture change
77° F.	14	2.4 — 1.7%
85° F.	29	1.7 — 1.3%
90° F.	20	1.3 — 0.9%

found no difference between non-sulfited powders reduced to 1% moisture by "cascade-conditioning" and those held at 77° F. for 220 days (15 correct in 42 total judgments, probability 0.410).

Packaging atmosphere. After storage for 90 days at 90° F. the informal panel was unable to detect differences between samples that had been vacuum- and air-packed, using both sulfited and non-sulfited powders. Under these storage conditions, it appears that vacuum packing does not offer any major improvement in flavor. However, it should be emphasized

that these are preliminary results based on a small number of samples.

Sulfur dioxide. Powders containing 120 p.p.m. (MFB) sulfite were obtained from concentrates with 220 p.p.m. (MFB) added sulfite. Sulfite treatment did not protect pineapple juice from flavor damage at higher drying temperatures as was found with tomato juice *(3)*. The informal group of judges detected scorched flavors in freshly prepared sulfited as well as non-sulfited powders dried at 160° F. After cascade-conditioning of powder dried at 150° F., and storage at 90° F. for 90 days, the informal taste judges preferred sulfited to non-sulfited powder. These preliminary results suggest that sulfite has a protective effect during storage. However, these same judges agreed that both sulfited and non-sulfited powders had developed scorched flavors within 30 days at 100° F. The length of time samples can be stored at various temperatures without developing off-flavors and any increase in these storage times as a result of sulfite treatment have not been determined precisely.

Color. The powder is pale yellow in color as freshly prepared. Water-soluble color on the fresh powder is expressed as 63 in Klett units as noted above. Samples stored at 100° F. for 30 days showed a soluble-color reading of about 120 Klett units, and these samples were obviously poor in flavor as unanimousy noted by an informal panel of 12 judges. Samples stored at 70° F. for 12 months had a soluble color of 67 Klett units; those stored at 77° F. for 8½ months had a soluble color of 64 Klett units. This indicates that the color changed very little at the lower storage temperatures.

Ascorbic acid. The ascorbic acid in the concentrate (47 mg. per 100 g.) was not changed by the drying process. Samples stored at 70° and 90° F. for 6 months showed no decrease in ascorbic acid, but 100° F. sample sshowed a 50% decrease. Sulfite was not effective in preventing loss of ascorbic acid.

SUMMARY

Pineapple-juice powder of good quality is produced from commercial concentrate by drying at 150° F. at 2 mm. Hg pressure for 2 hours and 45 minutes in a vacuum shelf drier. The product is packaged with an in-package desiccant and stored at progressively higher temperatures up to 90° F. as the moisture content of the powder decreases. The addition of SO_2 to the concentrate protected the flavor of powders stored at 90° F. If the product is stored at temperatures of 70° to 77° F., there is no need for addition of SO_2 or other preservative, since the flavor and color changes after one year are negligible.

Under conditions of the experiments reported here, vacuum packaging was not found necessary for retention of flavor.

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Effect of Various Freezing Methods on Quality of Poultry Meat[a]

(Manuscript received December 30, 1957)

W. W. Marion[b] and
W. J. Stadelman
Purdue University, Lafayette, Indiana

Commercial use of air blast freezing and liquid freezing have made possible very rapid freezing of meats. Both methods are being used with poultry meat. Whether the process is considered from the standpoint of preserving living cells and tissues or in the preservation of meat, freezing appears to be a single physical event whereby pure water is removed from solution and isolated into ice crystals. Ultimate size and rate of crystal growth are temperature dependent and their control is primarily a problem in heat exchange *(6)*. Several investigators *(7, 8, 9, 10)* have reported upon aspects of freezing and storage of poultry meat in relation to its subsequent quality with particular reference to percentage drip, resulting from the thawing process, total cooking loss, and palatability.

The primary purpose of this study was to compare the effect of four methods of freezing on three criteria of poultry meat quality: percentage drip, percentage total cooking loss, and tenderness of the *pectoralis major* muscle. The second objective was to conduct a survey on the color of frozen turkey and determine the color most desired by the public.

PROCEDURE

The four freezing methods are described as follows:

1. Liquid freeze unit in which a 26% NaCl solution by weight was used as the cooling medium. Brine temperature was adjusted to 10° F. (−12° C.) and rate of flow maintained at 50 feet per minute.
2. Liquid freeze unit, as described above, for 15 minutes after which the poultry was transferred to a plate-type, still-air household freezer.
3. Complete freezing in the plate-type household freezer.
4. A commercial freezer adjusted to −15° F. (−26° C.) with air circulating at the rate of 150 feet per minute.

A preliminary test showed that the internal temperature of turkey fryers, averaging 6¼ pounds, decreased from 35° F. (2° C.) to 20° F. (−7° C.) in about 100, 435, 600 and 435 minutes for methods 1, 2, 3 and 4, respectively.

Four experiments were conducted during the first study. The first class of poultry frozen by the various methods was 11-week-old White Rock-Cross fryers. These 40 fryers, as in all other instances, were killed by externally severing the carotid arteries, scalded for 45 seconds in 140° F. (60° C.) water, eviscerated and aged in slush ice for 24 hours. Upon removal from the slush ice, the fryers were drained and weighed to the nearest gram. Polyvinylidine bags were used for individually packaging the fryers. Each fryer was randomly assigned to a freezing treatment. Following freezing, the fryers were stored in a −5° F. (−21° C.) still-air freezer for one to 2 weeks prior to thawing and cooking.

Defrosting was done at a room temperature of approximately 80° F. (27° C.) for 12 hours. The poultry was retained in the polyvinylidene bags during defrosting. Percentage drip was calculated from the difference between the initial cooled weight and the weight taken following thawing and draining. Percentage total cooking loss was based on the weight loss of the breast section only which was removed and cooked in a hydrogenated animal fat at 325° F. (163° C.) for 10 minutes.

Tenderness of the *pectoralis major* muscle was measured by the Kramer Shear Press *(5)* in pounds of shear per gram of meat. The sample of muscle measured 2½ x 1¼ x ½ inches. Measurements were made on both the left and right major breast muscles after an hour of cooling in a 35° F. (2° C.) cooler, except where a delay of 24 hours prevented the completion of testing one-half of the samples. Untested samples were kept wrapped in aluminum foil during this time.

Forty White Rock-Cornish Cross yearling fowl were tested in the second experiment. Cooled weight of these fowl averaged 1850 g. All methods were similar to those outlined for the first experiment except time and method of cooking. Fowl were cooked in a reel-type oven at 340° F. (171° C.) for 2½ hours. Each hen was wrapped in aluminum foil prior to cooking.

Forty Broad-White turkey fryers, or 10 per freezing treatment, were tested in the third experiment. These fryers were 13 weeks of age and their weight after evisceration averaged about 1800 g. Methods used in previous experiments were used again here, except cooking time in a reel-type oven was reduced to 2 hours.

The final experiment utilized 16 mature Broad-Breasted Bronze turkey males. Average cooled weight of these turkeys was 10,000 g. Methods differed from the second and third experiments only in that the entire breast section was removed, wrapped in aluminum foil, and roasted for 3 hours. The average breast weight was 3300 g.

Data from these experiments were analyzed statistically by analysis of variance technique. Tests were made at the 0.05 level of probability.

To obtain the desired colors of frozen turkeys, in the second study, 16-week-old Broad white males were frozen at 0° F. (−18° C.), 10° F. (−12° C.) and 20° F. (−7° C.) in an immersion-type freezer in which the freezing medium was a 26% sodium chloride solution. These methods were labeled "A," "B" and "C," respectively. The two slowest methods of freezing were a commercial locker plant ("D") operating at −10° F. (−23° C.) with air circulation of 50 feet per minute and a plate-type, still-air freezer ("E") adjusted for −5° F. (−21° C.). Color of the frozen turkey is a function of rate of freeze; hence method "A" produced the whitest turkey and method "E" the darkest. A definite attempt was made to select turkeys of similar size and conformation, thereby forcing decisions to be made on color only. All turkeys were packaged in polyvinylidine film, evacuated and heat shrunk prior to freezing.

In cooperation with the Indiana State Poultry Association one turkey from each freezing method was placed on display at the Indiana State Fair. Visitors passing the displayed turkeys were requested to fill out a questionnaire indicating their first and last choices. Additional information was requested regarding a rough approximation of the age of the individual (either above or below 18 years of age), sex and size of community in which he or she lived.

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[a] Journal paper No. 1217 of the Purdue Agricultural Experiment Station.

[b] Present address: Poultry Husbandry Department, Iowa State College, Ames, Iowa.

RESULTS AND DISCUSSION

Method of freezing did not significantly affect percentage drip as measured in these experiments. Percentage drip for chicken fryers ranged from 5.3 to 5.8; fowl, 5.2 to 5.6; turkey fryers 4.3 to 5.3 and mature turkeys, 2.3 to 3.0. These values were larger in most instances than those reported in the literature *(3, 8, 10)* with the exception of one report *(9)* where there is close agreement in percentage drip obtained from turkeys cooled in slush ice similar to turkeys tested herein in the fourth experiment.

Differences in cooking losses between the freezing methods were found to be non-significant. The range in cooking losses for chicken fryers was 27.2 to 29.1; fowl, 32.9 to 33.8; turkey fryers, 22.5 to 25.0 and mature turkeys, 27.9 to 29.1. These findings agree with previous work *(9)* which indicated that rate of freeze did not significantly affect cooking losses. Tenderness values given in pounds of shear per gram of sample required to cut through a section of the *pectoralis major* muscle measuring $2\frac{1}{2} \times 1\frac{1}{4} \times \frac{1}{2}$ inches are shown in Table 1. Method of freezing did not significantly affect tenderness of the meat. Likewise, there were no significant differences between measurements on left and right muscles, except in the case of chicken fryers. Tenderness values for the *pectoralis major* muscle on the right half of the fryers were significantly higher indicating less tenderness than that found for the left half. This difference is unexplainable unless some delay in taking part of the measurements exerted an effect here. Even so, it would appear that both left and right muscles would be affected similarly.

A total of 1383 responses was received from individuals regarding the color preference study. The distribution of their choices relative to the most and least desirable colors of turkey is shown in Figure 1. The darkest colored turkey ("E") was selected by approximately 300 individuals as being their first choice. Slightly more persons chose "D" turkey (next to the darkest) as their first choice. There was a step-wise decrease in the number of responses favoring "C" and "B" turkeys. The whitest turkey ("A") appeared to be one of the most popular. The total number of responses equaled those for "D" turkey. These results indicate that this group of people might have a greater tendency to select one of the darker turkeys, possibly due to custom. If individuals liked the white turkeys, however, there was a definite tendency to select the whitest one. Comments from those who selected "A" turkey as first choice indicated that the white color connoted "cleanliness" or "sanitation."

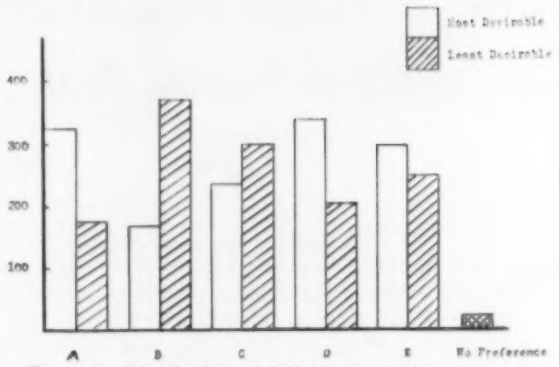

Figure 1. Distribution of individuals selecting the various turkeys; colors ranging from light (A) to dark (E).

Community size was divided as follows: over 100,000, 10,000 to 99,000, under 10,000 and farm; the distribution of responses was 389, 353, 249 and 392 for the community sizes, respectively. A trend similar to that previously described was found in each category except in cities of less than 10,000 population in which there was considerable increase in the number of individuals preferring "D" turkey as their first choice. Little deviation in the preference pattern was found when the data were reclassified according to age and sex of the individuals.

TABLE 1

Shear values of left and right pectoralis major muscles for kinds and classes of poultry as influenced by method of freezing. (Lower values indicate more tenderness).

Kind and class of poultry		Shear values (lbs./g. of meat) by freezing method			
		L.F.[1]	L.F. and A.[2]	A.[3]	C.[4]
Chicken fryers	Left side	9.2±1.9	8.6± .9	9.2±1.3	8.6±1.9
	Right side	10.6±3.0	9.1±2.7	10.0±2.3	10.4±2.6
Fowl	Left side	9.9±5.1	8.8±3.7	8.6±2.4	8.8±2.2
	Right side	10.1±4.8	9.1±3.3	8.4±3.2	8.0±1.6
Turkey fryers	Left side	7.5±2.7	7.2± .8	7.5±2.0	6.5±1.5
	Right side	7.5±3.2	7.5± .7	7.7±2.1	6.0±1.1
Mature turkeys	Left side	13.0±2.7	14.9±4.3	13.1±1.9	11.9±1.7
	Right side	11.4±1.8	15.0±2.2	13.3±1.5	12.4±5.4

[1] L.F. = Liquid freezer.
[2] L.F. and A. = Liquid freezer and Amana.
[3] A. = Amana.
[4] C. = Commercial freezer.

SUMMARY

Chicken fryers, fowl, turkey fryers and mature turkey toms were frozen by four different methods including liquid, plate, and moving air; the subsequent quality of meat was evaluated by physical measurements of percentage drip, percentage total cooking loss, and tenderness of the *pectoralis major* muscle.

Method of freezing was found to exert no significant effect on either of the measured variables; percentage drip, percentage total cooking loss, and tenderness of breast muscle. In the case of chicken fryers, there was a significant difference in tenderness between left and right *pectoralis major* muscles.

A larger number of individuals selected one of the two darkest colored turkeys as first choice in the color preference study. There was greater rejection of the lighter colored turkeys except for the whitest one which was one of the most frequently selected turkeys. Its very white color connoted to some individuals additional "cleanliness" or "sanitation."

Acknowledgment

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Bacteriological Content of Marketed Precooked Frozen Foods in Relation to Public Health

A. Doreen Ross and F. S. Thatcher[1]

Food and Drug Directorate, Department of National Health and Welfare, Ottawa, Canada

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A PRELIMINARY SAMPLING has been made of the prevailing bacteriological content of precooked frozen foods as purchased from retail markets in a Canadian city. The purpose was to assess the likelihood of any public health hazard arising from the widespread use of these foods and to provide some indication of the effectiveness of sanitation practices in the source factories. The foods examined were the products of nine different manufacturers from both the United States and Canada and included fifteen different kinds of the foods popularly known in Canada as "TV dinners," meat, fish and poultry pies, and cooked frozen dessert pies.

METHODS

In order to facilitate sampling of the hard frozen products that were held at −20° C., specimens were removed to a refrigerator at 5° C. for 10 to 12 hours to allow partial thawing. (Preliminary tests had shown that this treatment did not significantly influence the numbers of microorganisms recovered, but greatly improved the ease of preparation.)

A 50 g. composite portion of each specimen was removed aseptically and comminuted with 450 ml. of sterile distilled water in a sterile Waring blender. From the resulting dilution of 1:10, decimal dilutions to 1:10,000 were prepared, using dilution "blanks" at 5° C.

A further composite sample of 10 g. was added aseptically to 90 ml. of yeast-extract-veal-infusion broth and incubated for 18–24 hours as an enrichment culture prior to subsequent tests for the presence of salmonellae *(6)*.

Aliquots from the decimal dilutions were plated by standard methods using media appropriate for the purposes described below. Unless otherwise stated, the media were "Difco" products. (a) Standard plate count determinations were made using tryptone-glucose-extract agar incubated at 37° C. for 48 hours. (b) *Escherichia coli* was enumerated by the use of violet-red-bile agar incubated at 37° C. for 24 hours. Choice of this particular method was based on results of a preliminary study to compare the numbers of *E. coli* recovered by the respective use of violet-red-bile agar and standard M.P.N. procedures, with confirmation in each instance by IMViC tests. With the type of food under test, the more costly and time-consuming confirmed M.P.N. procedure allowed no greater accuracy of recovery of *E. coli* than did the direct plating method, provided that care was taken to record only typical red colonies surrounded by a precipitate of bile and to adhere to the 24-hour incubation period. (In this study, atypical colonies without the bile precipitate were found to be mainly *Flavobacterium* but smaller numbers of *Proteus*, *Aerobacter*, *Pseudomonas* and paracolon organisms also appeared on the plates. A random selection of 20 "typical" isolates were all *E. coli* in accord with IMViC tests). (c) Enterococci were determined by the use of Enterococcus Confirmatory Agar using 30 ml. per plate and incubating for 48 hours at 45° C. Earlier experience had shown too little advantage to justify the use, for survey purposes, of a sodium azide enrichment broth prior to plating, nor in using penicillin in conjunction with this medium, though the value of enrichment is recognised for specimens containing small

[1] Microbiology Section, Food and Drug Directorate, Department of National Health and Welfare, Ottawa, Canada.

numbers of enterococci. A group of 12 isolates from this medium had previously been shown to be streptococci of group D, Lancefield. (d) Staphylococci were isolated by the use of phenol-red-mannitol-salt-agar incubated at 37° C. for 48 hours, followed by determination of the ability to produce coagulase by five mannitol-positive colonies per specimen using "Difco" rabbit plasma as the test substrate. (e) Salmonellae were determined by inoculating selenite broth with the yeast-extract-veal-infusion enrichment culture previously described. After incubation at 37° C. for 18 hours the selenite preparation was used to streak plates of Bismuth Sulphite Agar and SS Agar. Representative colonies among those resembling *Salmonella* were used to inoculate Urea Agar, Triple Sugar Iron Agar, Simmons' Citrate Agar, gelatin, 1% tryptone broth for production of indole, MR-VP Medium, semi-solid veal-infusion agar for the detection of motility and Moeller's Cyanide Broth *(1)*. Cultures having appropriate reactions for *Salmonella* at this stage were tested with Polyvalent-O-*Salmonella* diagnostic sera and for the production of acid or gas in dextrose, lactose, sucrose, adonitol, dulcitol, inositol, mannitol, salicin and xylose broths. These tests allowed the distinction between salmonellae and members of the paracolon group. These latter organisms were subdivided and classified as members of the Bethesda-Ballerup Group, Providence Group, and the Arizona Group after Edwards *(1)*, the last being subject to confirmation with appropriate typing sera. The determination of the presence of the various paracolon groups was undertaken in response to the recent review by Edwards *et al. (2)* on the established pathogenicity of some of these organisms, particularly those of the Arizona group.

A selection of 31 of the more heavily contaminated food specimens were heated in accord with the manufacturers' recommendations, and the surviving populations determined by the same series of tests as described above.

RESULTS

Average values for the microbial determinations made from each type of food are listed in Table 1. It may be noted that the classical indicators of fecal contamination, *E. coli* and enterocci, were commonly present. Space requirements preclude the tabulation of all individual determinations, but *E. Coli* was found in 54 specimens in numbers ranging to 130,000 per g.; enterococci were present in 60 specimens with a maximum count of 140,000 per g. Representatives of the Bethesda-Ballerup group of paracolon bacteria were found in 14 specimens, the Providence Group in 2. Representatives of the Arizona group and of *Salmonella* were not found. Coagulase-positive staphylococci were determined in 21 specimens ranging in numbers to 70,000 per g.

Table 2 lists the minimum, median, "three-quarter" and maximum values for each of the numerically determined bacterial categories. (The "three quarter" value is that below which 75% of all specimens would conform). Clearly the last quartile represents very heavily contaminated specimens, and offers an extreme contrast to the bacteriological values that can be attained commercially as shown by the specimens in the first quartile. This applies particularly to the staphylococci and enterococci which at the "75%" point occur in the respective numbers of 1,000 and 256 per gram.

Table 3 lists the numbers of bacteria in the several test categories that survived heating of the food specimens in an oven in accord with the manufacturers' recommendations, which ranged from 400° F. to 450° F. and for times ranging from 20 to 40 min. Staphylococci and enterococci each survived in 10% of the specimens. The degree of reduction in bacterial numbers in the various specimens as expressed by the standard plate count ranged from 57.5% to 100%, with an average reduction of 99.9%. The maximum numbers of bacteria of the various test categories to survive heating and the respective initial values from the same specimens were as follows: standard plate count 85,000 and 200,000 per g.; enterococci 1,900 and 6,000 per g.; staphylococci 150 and 1200 per g. *E. coli* and other members of the Enterobacteriaceae failed to

TABLE 2

Some distribution values for bacteria of specified "indicator" categories in 117 market specimens

Bacterial indicator	Bacterial values: Number per gram			
	Minimum	Median	"¾ Value"	Maximum
Plate Count	50	13,000	50,000	22×10^6
E. coli	0	0	15	130,000
Enterococci	0	5	256	140,000
Staphylococci	0	180	1,000	70,000

TABLE 1

The bacteriological content of frozen precooked dinners and pies

Index No.	Manuf.	Specimen	No. of specimens	Country of origin	Bacteriological Findings [1]					
					Average No. of organisms per gram				No. of specimens containing	
					S.P.C. ($\times 10^3$)	*E. coli*	Enterococci	Staphylococci	Coagulase pos. staphy.	Paracolon organisms
1–7	A	Chicken Dinner	7	U.S.	24	50	3.4	1825	1	1
8–14	A	Turkey Dinner	7	U.S.	90	444	295	861	1	2
15–21	B	Beef Dinner	7	Can.	4	21	8.5	226	2	4
22–24	A	Chicken Pie	3	U.S.	14	1.7	11.7	65	0	2
25–31	C	Salisbury Steak	7	U.S.	740	33	.7	490	0	3
32–38	C	Swiss Steak	7	U.S.	30	0	38	18	2	0
39–44	D	Steak and Onions	5	Can.	113	2	48	690	1	0
45–49	D	Fillet of Sole	5	Can.	68	1	2510	519	0	1
50–54	E	Turkey Pot Pie	5	U.S.	213	0	8300	15,620	2	2
55–59	E	Chicken Pot Pie	5	U.S.	430	3	3250	12,200	2	3
60–64	E	Beef Pot Pie	5	U.S.	5	3	0	14	0	3
65–69	F	Chicken Pie	5	U.S.	7	4	59	526	1	4
70–74	F	Beef Pie	5	U.S.	11	8	1356	1384	0	1
75–77	F	Turkey Pie	3	U.S.	4	1.7	177	579	0	2
78–80	G	Pork Pie	3	Can.	8	10	0	1350	0	2
81–83	G	Lamb Pie	3	Can.	15	0	1.7	65	0	2
84–86	G	Veal Pie	3	Can.	16	0	1.7	433	0	1
87–89	G	Chicken Pie	3	Can.	12	108	43	633	1	0
90–92	G	Turkey Pie	3	Can.	10	568	18	120	0	1
93–95	G	Beef Pie	3	Can.	3	0	0	237	0	3
96–98	H	Beef Pie	3	Can.	7,525	43,500	48,550	334,000	1	0
99–101	I	Chicken Pie	3	Can.	13	7	123	420	2	1
102–104	I	Beef Pie	3	Can.	12	7	0	1500	2	2
105–107	I	Steak and Kidney Pie	3	Can.	721	100	1450	263	3	3
108–112	E	Pumpkin Pie	5	U.S.	12	738	1102	38	0	0
113–117	E	Cocoanut Pie	5	U.S.	1	0	4	26	0	0

[1] The presence of *Salmonella* could not be confirmed in any sample.

TABLE 3

The survival of specified bacteria after cooking in accord with manufacturers' specifications[1]

Bacterial category	Range of reduction in bacterial numbers after cooking	Specimens showing survival of bacteria	Maximum No. of bacteria surviving in individual specimens
	(%)	(%)	(No./g.)
St. Plate Count	57.5–100	77	85,000
E. coli	100	0	0
Enterococci	67.5–100	10	1,900
Staphylococci	85–100	10	150

[1] 400° F. to 450° F. for 20 min. to 40 min. depending on manufacturer.

survive the heating process even though a maximum initial value of 130,000 *E. coli* per g. had been present.

DISCUSSION

The data presented suggest to the authors that a substantial proportion of the foods examined contained numbers of bacteria sufficiently high to indicate a probability of quality deterioration, and established an objectionably high degree of contamination from fecal and naso-pharyngeal or suppurative sources. Contamination of this order might well prove to be sufficient in some instances to denote a risk of enteric infection or of staphylococcal food poisoning in the event that the foods did not attain the recommended temperatures during cooking or through failure to maintain consistent freezing pending sale or after purchase. Both conditions of treatment are suspected of occurring frequently.

It is possible that some of the specimens containing the higher numbers of bacteria may have been subject to transient thawing either during transport or while exposed for retail sale.

The coagulase-positive staphylococci found in these foods may well have originated from human sources of contamination. *E. coli* and the paracolon group might arise from either human or animal sources, the latter origin being particularly suspect in foods containing poultry meats.

The fact that fault in prevailing practice by retailers and the housewife may contribute to multiplication of initial contamination would seem to place a weighty onus on the manufacturer to reduce contamination to a practicable minimum by strict supervision of all aspects of hygiene in the production of these foods. This is a conclusion expressed by other investigators *(3, 4, 5, 7)*. However, in the light of infrequent recording of illness from pre-cooked frozen foods, further study is required to determine the precise values for specific "indicator" categories of bacteria that would be commensurate with such practicable minima.

SUMMARY

Bacteriological examination of 117 specimens of precooked frozen foods purchased from retail outlets has established that a proportion of these foods are highly contaminated. Certain specimens were shown to contain *E. coli*, coagulase-positive staphylococci and enterococci in large numbers. Members of the Bethesda-Ballerup and Providence groups of the paracolon organisms were found. The requirement of exacting standards of hygiene in the source factories was deduced.

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The Influence of Added Monosodium Glutamate on the Flavor of Processed Green Beans[a]

Lois A. Sather, L. A. Pettitt,[b] and R. W. Hirzel[c]

Department of Food and Dairy Technology, Oregon State College, Corvallis, Oregon

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Use of monosodium glutamate (hereafter MSG) as a seasoning agent in canned and frozen green beans has been investigated by several laboratories. Norton, Tressler and Farkas *(4)*, Shuey and Eichenberger *(5)*, and the Glutamate Manufacturers' Technical Committee *(2)*, reported an improved or better flavor when a given per cent of MSG was added during processing or cooking. Van Duyne, Charles, Titus and Wheeler *(8)* were unable to show an improvement in flavor when MSG was added to frozen green beans. With respect to canned green beans, Girardot and Peryam *(1)*, Sjöström and Crocker *(7)* and the Glutamate Manufacturers' Technical Committee *(2)*, reported an improved or better flavor when MSG was added to the canned product.

All but one of the above investigators used panels of 4 to 10 trained or experienced tasters for evaluating the organoleptic properties of green beans containing added MSG. Girardot and Peryam *(1)* recorded 80 judgments from a large panel of untrained tasters on canned green beans.

There is a distinctive flavor difference between pole and bush green beans and, since the above investigators did not state the type of bean used, it was deemed advisable to grow both types of beans under the same environmental situation and to test the effect of added MSG on the canned and frozen beans. Further, it appeared of importance to determine if a large group of untrained or inexperienced tasters could detect an improvement in the flavor of beans to which MSG had been added prior to processing.

EXPERIMENTAL

Canned and frozen one-inch cut and frozen french cut green bush beans and green pole beans, and canned 1½ inch cut wax bush beans were packed under the authors' supervision during August, 1955, at commercial processing plants in Oregon. The Processor variety of bush beans, the FM-1 variety of Blue Lake pole beans and the Puregold variety of wax beans were used. Samples of each type and cut of bean were processed containing no added MSG, a low level of added MSG (approximately 0.125% by weight) and a higher level of added MSG (approximately 0.20% by weight). All samples were prepared and processed by commercial procedure. Samples were not preselected for any given quality factor but care was taken that the treated and untreated samples of any given lot were from the same source of raw material. In the frozen samples just prior to overwrapping and freezing, a water solution of MSG was sprayed over the top of the beans in the package by an International liquid dispenser. In the canned samples, a water solution of MSG was added after salt and beans had been measured into the cans just prior to filling the cans with hot water.

The 4 lots of frozen beans were stored at 0° F. (−18° C.). Canned wax bush beans were stored at 70° F. (21° C.). Canned green bush beans and the canned green pole beans were divided and half of each lot stored at 70° F. (21° C.) and the other half at 100° F. (38° C.).

The Hunter Color Difference Meter was used for making color readings on 4 replicate samples from each treatment of each lot of beans after approximately 3, 6, and 14 months' storage. The sample spinning technique developed by Sidwell and Cain *(6)* was used. The beans were placed in the plastic dish at random and the Hunter "Rd," "a" and "b" readings obtained and recorded. For the green beans, the instrument was standardized to the values for the SKC-15 "Kitchen Green" color plaque supplied by the National Bureau of Standards. For the wax beans, the SBC-35 plaque was employed.

Mr. Edmund B. Adamic, Research Chemist, International Minerals and Chemical Corporation, analyzed the canned and frozen samples for 1-MSG · H_2O content.

Flavor preference tests were made on each lot of bean samples after approximately 3 and 6 months' storage. After approximately 14 months' storage, flavor preference tests were made on only the canned pole beans and bush beans stored at 70° F. (21° C.), canned pole beans stored at 100° F. (38° C.), and the frozen one-inch cut pole beans.

On all flavor tests, Oregon State College students served as tasters. The tasters were not screened for tasting acuity but were hired on a "first come" basis following a notice given to all students during college registration. The tasters were allowed to taste anytime between 8:30 and 11:30 a.m. and 1:30 to 4:30 p.m. The number of tasters in each test ranged from 94 to 142.

At each testing period, a multiple comparison, reference-preference type flavor test was used. The general procedure for conducting this type of test was: Samples were served in coded paper cups on a metal serving tray to tasters seated in isolated testing booths. Each taster at each testing period received 4 sample cups. One cup, labeled "R," contained an untreated sample as reference. The other 3 cups, labeled with 3 digit random numbers, contained the 2 glutamate-treated samples and an untreated sample. The tasters were asked to indicate, on a printed ballot, preference for each of the 3 coded samples in direct relation to the reference sample. The printed ballot provided the following 7-point scale on which to score the samples:

(7) Much better flavor
(6) Moderately better flavor
(5) Slightly better flavor
(4) Neither better nor poorer flavor than reference
(3) Slightly poorer flavor
(2) Moderately poorer flavor
(1) Much poorer flavor

Score points did not appear on the ballot but were assigned to the individual judgments prior to statistical interpretation. All flavor data were analyzed by analysis of variance.

The ballot also instructed the tasters to "Rinse mouth with tap water before tasting each sample." This precaution was taken in order to minimize any possible carry-over flavor effect.

[a] Submitted as Technical Paper No. 1094, Oregon Agricultural Experiment Station.

[b] Present address: Home Economics Department, University of Utah, Salt Lake City, Utah.

[c] Present address: 925 West Michigan, Lansing 33, Michigan.

On the 6 months' storage tests, the canned pole beans stored at 70° F. (21° C.), were also tested by the paired-comparison preference method. This method consisted of serving the tasters 2 coded samples, one untreated and the other containing MSG and asking them to record on a written ballot which sample they preferred and why.

On the 14 months' storage tests, the 4 lots of samples tested by the reference-preference method were also tested by the paired comparison method. Also each lot of samples was tested twice by the reference-preference method, once with the samples arranged in random order on the serving tray, and again with samples arranged in fixed order on the serving tray from no MSG first to the higher level of MSG last. To alleviate any possible effect of testing day, one lot of samples was served on 4 consecutive testing days. Each day one-fourth of the tasters scored the samples by one of the 4 testing methods: reference-preference fixed order, reference-preference random order, paired comparison, untreated and low level of MSG, and paired comparison, untreated and higher level of MSG. Each taster scored the beans by each of the 4 methods over the 4 days. The score of any taster who was absent for any test was not included in the final analysis.

For serving, the canned beans were heated to boiling and then set over hot water to keep warm. New cans were opened and heated every 30 minutes during the testing period. Two 10-ounce packages of frozen beans were always cooked in the same pan at the same time. The 2 packages of frozen beans were added to 8 ounces of boiling water containing 40 grains of salt and simmered until tender, approximately 15 minutes after the water returned to a boil. Preliminary experiments on the frozen samples indicated that this salt level was approximately the same as added by the commercial plants to the canned samples.

RESULTS AND DISCUSSION

No differences in color between the glutamate treated and the untreated canned and frozen green beans and wax beans were evident after 3, 6, and 14 months' storage.

The per cent by weight 1-MSG · H_2O in the processed beans was in most cases lower than the original expected values of 0.125% and 0.20% (based on added amounts). As there was no significant loss of MSG from the samples during storage, the lower recovery might have been due to the probable errors of sampling and analysis. Also, in the canned samples there was a loss of some of the liquid from the cans during the sealing operation. The total per cent by weight of 1-MSG · H_2O in each lot of beans is given in Table 1.

The mean scores of the 3 and 6 months' flavor tests are given in Table 2.

After 3 months' storage of the beans, the student panels indicated no flavor preference between glutamate-treated and untreated beans from any lot except the canned green bush beans stored at 100° F. (38° C.). A group of 138 tasters indicated a slight flavor preference for canned green bush beans stored at 100° F. (38° C.) containing either the low or high level of MSG as compared to the untreated sample.

TABLE 1

The percentage by weight 1-MSG·H_2O in processed bean samples

	No addition	Low level	Higher level
Frozen french-style bush beans	0.010	0.120	0.185
Frozen one-inch cut bush beans	0.020	0.080	0.145
Canned green bush beans	0.030	0.115	0.145
Frozen french-style pole beans	0.020	0.120	0.170
Frozen one-inch cut pole beans	0.020	0.135	0.195
Canned green pole beans	0.045	0.090	0.165
Canned wax bush beans	0.060	0.145	0.230

After 6 months' storage, a group of 120 tasters indicated a slight flavor preference for canned green bush beans stored at 70° F. (21° C.) containing the low level of MSG over the sample containing no added MSG. A group of 133 tasters indicated a slight flavor preference for frozen one-inch cut green pole beans containing the low level of MSG over samples containing no added MSG and samples containing the high level of added MSG. On no other lots of beans were any flavor preferences indicated at this testing period. The results obtained from the paired comparison and reference-preference tests on canned green pole beans were in agreement, with no preference indicated for any of the samples by either testing method.

Results of the 14 months' flavor tests are given in Table 3.

On the 14 months' storage tests, the random order reference-preference, fixed order reference-preference, and paired comparison tests employed on 4 lots of green beans gave the same results in all instances except one. On canned bush beans stored at 70° F. (21° C.), the lower level of added MSG was preferred over the sample having no addition of MSG when scored by the paired comparison test. This preference was not indicated on the reference-preference tests. No preferences were indicated for any other samples tested.

Analysis of the reference-preference tests indicated that there was a significant taster variation. Therefore, results of these tests were also analyzed for dif-

TABLE 2

Mean flavor scores of reference-preference flavor tests after 3 and 6 months' storage

	3 months				6 months			
	N[1]	O[2]	Low[3]	Higher[4]	N[1]	O[2]	Low[3]	Higher[4]
Frozen french-style green bush beans	142	4.0	3.9	4.1	118	4.2	3.8	3.9
Frozen one-inch cut green bush beans	139	4.1	4.0	3.7	106	3.9	3.8	3.7
Canned green bush beans, 70° F. storage	116	4.0	3.9	4.0	120	3.9	4.3[5]	4.1
Canned green bush beans, 100° F. storage	138	3.8	4.3[5]	4.1[5]	94	3.8	4.1	3.7
Frozen french-style green pole beans	127	4.4	4.2	4.1	113	3.9	4.2	3.9
Frozen one-inch cut green pole beans	137	4.1	4.0	4.1	133	3.8	4.1[5]	3.6
Canned green pole beans, 70° F. storage	141	4.0	3.9	4.0	116	3.9	4.1	4.2
Canned green pole beans, 100° F. storage	138	3.9	4.0	4.0	106	3.6	3.7	3.6
Canned wax bush beans, 70° F. storage	140	3.9	4.1	4.2	108	3.8	4.0	4.0

[1] N = Number of tasters.
[2] O = Same as reference sample. No added MSG.
[3] Low = Approximately 0.125% MSG.
[4] Higher = Approximately 0.20% MSG.
[5] Significantly better (.05 level) than the untreated sample.

TABLE 3

Mean scores of reference-preference flavor tests and the number of tasters indicating a preference on paired comparison flavor tests after 14 months' storage

Type of flavor test	Canned pole beans 70° F. storage 139 tasters[1]			Canned bush beans 70° F. storage 130 tasters			Canned pole beans 100° F. storage 137 tasters			Frozen 1 in. pole beans 0° F. storage 126 tasters		
	No MSG	Low MSG	High MSG	No MSG	Low MSG	High MSG	No MSG	Low MSG	High MSG	No MSG	Low MSG	High MSG
Reference-preference, fixed order	4.13	3.36	3.96	4.08	4.32	4.29	3.89	3.60	3.97	3.78	3.86	3.68
Reference-preference, random order..	4.05	4.00	4.16	4.06	4.39	4.33	3.88	3.96	3.99	4.21	3.98	3.92
Paired comparison, O and low.....	72	58		47	81[2]		60	73		55	67	
Paired comparison, O and higher.....	74		62	57		69	58		73	68		55

[1] On the paired comparison tests the number of tasters indicating a preference does not equal the total number of tasters as some tasters indicated no preference.

[2] Significant at the 5% level.

ference by compiling the slightly poorer and slightly better scores as meaning slightly different flavor; the moderately poorer and moderately better scores as meaning moderately different flavor; and the much poorer and much better scores as meaning much different flavor. When analyzed in this manner, it was evident that the individual tasters were detecting a difference in flavor between the MSG treated and the untreated beans but as a group did not agree as to which samples were preferred.

Further research on canned green beans containing 0 and 0.15% MSG which utilized a home panel of 220 families supports these conclusions. This research which compares the preference results obtained from the panel of 130 to 180 students and a 220 family home panel will be reported in full later.

SUMMARY

After approximately 3, 6, and 14 months' storage, color, and flavor evaluation tests were made on canned and frozen bush and pole green beans and bush wax beans containing no added MSG, a low level of added MSG (approximately 0.125% by weight) and a higher level of added MSG (approximately 0.200% by weight). All samples were prepared by commercial procedures. In the canned samples, MSG was added after salt and beans had been measured into the cans and in the frozen samples, MSG was added just prior to sealing the package. The beans were not preselected for quality but were regular plant run.

Readings from the Hunter Color Difference Meter indicated no differences in color between MSG treated and untreated canned and frozen green bush and pole beans and bush wax beans after 3, 6, and 14 months' storage.

Under the conditions of this test, large panels (94 to 142 tasters) of untrained student tasters indicated no consistent flavor preferences for the MSG treated or untreated bean samples.

The student tasters could detect a difference in flavor between the MSG treated and the untreated beans but could not as a group consistently agree on preference.

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Influence of Ripening Temperature, Ripeness Level, and Growing Area on Quality of Canned Bartlett Pears

L. L. Claypool, Sherman Leonard, B. S. Luh, and Marion Simone

Departments of Pomology and Food Technology, University of California, Davis, California

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FACTORS INFLUENCING quality of canned pears are of vital interest to fruit processors. The dessert quality of the canned product is related to the quality of the fresh fruit, which varies greatly with the variety, maturity at harvest *(1, 2, 7, 11, 18, 20)*, and the temperature to which the fresh pears are subjected while in transit, storage, and ripening *(3, 7, 11, 18, 20)*. The changes in chemical composition and texture of pears during post-harvest handling have been published by various workers *(5, 6, 7, 8, 14, 20)*. The importance of the emanation of volatile compounds during the ripening of pears *(9, 10)* to the flavor quality of the canned product has been reported *(13, 15)*. Ezell and Diehl *(7)* studied the relation of maturity and handling of Bartlett pears to quality of the canned product in the Pacific Northwest. They stated that pears harvested at a pressure test range of 15 to 17 pounds as measured by a Magness-Taylor fruit tester resulted in a higher quality finished product than fruits harvested at higher or lower pressure readings. A ripening temperature range of 70° F. (21° C.) to 75° F. (24° C.) gave the most rapid and uniform ripening and resulted in the best canned product. Pentzer *et al. (18)* recommended pears to be ripened at 60° F. (15.5° C.) to 70° F. (21° C.) for the best dessert quality. Tindale and Trout *(20)* reported that the minimum ripening temperature varies with the variety of pears, and that Williams Bon Chrétien (Bartlett) pears did not ripen below a temperature of 55° F. (13° C.) due to core breakdown and scald without production of adequate juice and flavor. Full color, flavor, and juice were best developed at 65° F. (18° C.) to 70° F. (21° C.). The flavor characteristics of the fruits used in previously mentioned studies were not evaluated by statistically sound sensory tests; the conclusions were drawn from observations of the experimenters.

In 1956, 5,427,000 cases of Bartlett pears were canned in California. This volume was about half of the total production of 11,117,000 cases in the United States in the same year. The trend in the California canning industry is changing from ambient ripening to that under controlled temperature and humidity to improve quality and to increase case yield. Because of the extreme interest in the factors influencing quality of canned Bartlett pears as expressed by growers and canners in California, experiments were designed in the 1954 and 1955 seasons to investigate the optimum conditions for post-harvest handling of the fresh pears for canning. In this report, the effect of ripening at 68° F. (20° C.), 77° F. (25° C.), and 86° F. (30° C.) on flavor, chemical composition and volatile reducing substances in canned California Bartlett pears is presented. The effect of growing area on quality of the canned product is also discussed.

EXPERIMENTAL MATERIALS AND METHODS

Bartlett pears. For the 1954 season, one-half ton of pears from an orchard at Lake County was used to investigate optimum ripening temperatures and ripeness levels for canning. In the 1955 season, one-ton lots of pears were harvested from commercial orchards in (1) Lake County on August 8; (2) Sacramento River District on July 28; and (3) Santa Clara County on August 12. An average pressure range of 16 to 17 pounds, when tested with the Magness-Taylor type pressure tester with a $\frac{5}{16}$ in. plunger, was used in both years as the standard for harvest maturity. The pears were hauled to Davis within 8 hours after harvest and stored at 32° F. (0° C.) for a minimum of 6 days and then ripened at 68° F. (20° C.), 77° F. (25° C.), and 86° F. (30° C.), respectively, under a relative humidity of 85%. They were then canned at different stages of ripeness as determined by the pressure test. Unless otherwise stated, the pears were canned as near as possible to 2 pounds average pressure test. The 0-10 pound tester was used for this latter determination.

Physical and chemical tests. Twenty pears were taken randomly from 160 lbs. for firmness and chemical tests. Pressure test, total titratable acidity, and soluble solids were determined on the fresh and canned product as described in a previous paper *(11)*. Volatile reducing substances, methyl alcohol, and carbonyl compounds in the canned pears were determined as described previously *(15)*, with the fruit and syrup combined.

Canning. The pears were peeled and cored in a Food Machinery and Chemical Corporation pear peeler and then dipped into tap water for 2 to 3 minutes. The halved pears were trimmed and sorted before canning. Eighteen oz. (±0.1 oz.) of pears and 11 oz. of 25° Brix sucrose syrup were added into each No. 2½ can. Segments of pear halves were used to obtain the precise fill weight of 18 oz. The cans were sealed under 15 inches of vacuum, cooked at 100° C. (212° F.) for 32 minutes in a rotary cooker, cooled rapidly in a rotary water cooler for 7 minutes and stored at room temperature for subsequent analyses.

Organoleptic evaluation. Three months after canning, the pears were evaluated for flavor quality by 9 or 10 judges, who were trained and selected for acuity and consistency in evaluating canned pears of varying quality. A balanced incomplete block design as described by Cochran and Cox *(4)* was applied. Four cans of each sample were used for each replication. The pears were diced with all the syrup added back, and then served in coded beakers under red lighting. The judges ranked three samples at a time for pear flavor quality, 1 being highest in natural pear flavor, and 3 being lowest. Analysis of variance was applied to the data, using the individual judge's total ranks for each sample as the variants. Least significant differences were determined by the Tukey method:

$$\text{LSD} = \text{tabular } q \times \sqrt{\frac{\text{mean square of error}}{\text{number of judges}}}$$

RESULTS

Effect of ripening temperature on pear composition and flavor quality. Differences in chemical composition among pears ripened at 68° F. (20° C.), 77° F. (25° C.), and 86° F. (30° C.) to a firmness of approximately 2 lbs. are shown in Table 1. Pears ripened at 86° F. (30° C.) were slightly higher in soluble solids than those ripened at 77° F. (25° C.) and 68° F. (20° C.). Ripening temperature variations resulted in little difference in acidity among the samples. In all 3 growing areas, pears ripened at 86° F. (30° C.) were lower in volatile reducing substances (VRS) content than those ripened at 77° F. (25° C.) and 68° F. (20° C.). This may result either from a change in the physiology of the pears due to temperature differences or an increase in the volatility of the VRS at higher temperatures, or both. Pears from the Sacramento River District were higher in total VRS content than those grown at Lake County. However, the effect of ripening temperature on VRS content of Sacramento River pears (Table 1, samples F, G, and H) was much less pronounced than those from the other two districts (samples B, C, D, and J, K, and L).

The results of sensory evaluation of Bartlett pears canned after ripening at different temperatures are illustrated in Figures 1 and 2. The statistical significance of these results was determined by analysis of variance (Tables 2 and 3). Samples ranked highest in natural pear flavor quality are indicated by the lower average rank. Figure 1 represents the result on the canned Bartlett pears from Lake County (1954) which were ripened at different temperatures to different pressure tests. Figure 2 shows the results of the 1955 pack on the effect of ripening temperature and growing area on the flavor of the canned product. The data indicate that more natural pear flavor in the

TABLE 2

Effect of ripening temperature and ripeness level on flavor quality—1954 pack

Analysis of variance			
Variation	Mean square	Least significant differences (probability level)	
		.05	.01
Ripening temperature	100.82***	1.0	1.2
Ripeness level	252.85***	1.5	1.8
Temperature x level	44.60***		
Error	3.90		

***Significant at the 0.001 probability level.

TABLE 1

Chemical composition of fresh and canned Bartlett pears ripened at various temperatures—1955 pack

Fresh fruit

Sample	Date	Ripening temperature	Average pressure test in lbs. (5/16 in. plunger)	Soluble solids as ° Brix at 20° C.	pH	Total acidity as per cent citric
Lake County						
*A	8/8	.....	17.0	13.3	4.00	0.192
B	9/19	68° F. (20° C.)	2.1	14.01	4.13	0.187
C	9/19	77° F. (25° C.)	2.0	14.73	4.18	0.204
D	9/19	86° F. (30° C.)	2.3	16.00	4.19	0.213
Sacramento River District						
*E	7/28	.....	16.1	10.60	4.00	0.250
F	8/9	68° F. (20° C.)	2.0	12.08	4.02	0.287
G	8/9	77° F. (25° C.)	1.8	11.20	3.80	0.256
H	8/9	86° F. (30° C.)	2.2	12.48	4.08	0.290
Santa Clara County						
*I	8/12	.....	16.3	10.30	4.10	0.136
J	9/6	68° F. (20° C.)	2.0	11.49	4.20	0.154
K	9/6	77° F. (25° C.)	1.9	11.50	4.25	0.155
L	9/6	86° F. (30° C.)	2.2	12.42	4.20	0.158

Canned fruit
(fruit and syrup combined)

Sample	Date	° Brix at 20° C.	pH	Total acidity as Brix/acid		Volatile reducing substances microequiv/ 100 g.	Carbonyl compounds as acetaldehyde (p.p.m.)
				% citric acid	Ratio		
Lake County							
*A	8/8	.....	.....	.....	.....	.....	.....
B	9/19	17.63±.25	4.30	.116±.004	153.1±5.5	510	3.4
C	9/19	18.23±.37	4.29	.118±.003	154.7±5.1	543	4.0
D	9/19	18.65±.17	4.26	.119±.003	157.0±5.5	374	4.0
Sacramento River District							
*E	7/28	.....	.....	.....	.....	.....	.....
F	8/9	16.35±.10	4.20	.162±.004	101.3±7.2	597	3.3
G	8/9	16.78±.07	4.18	.159±.005	105.4±3.4	564	4.2
H	8/9	16.93±.31	4.10	.157±.003	110.5±2.0	561	3.3
Santa Clara County							
*I	8/12	.....	.....	.....	.....	.....	.....
J	9/6	16.75±.44	4.40	.087±.004	189.2±2.0	590	6.7
K	9/6	16.20±.11	4.41	.090±.002	179.9±5.6	521	2.0
L	9/6	16.96±.10	4.46	.088±.003	193.5±6.7	443	2.2

*Samples at harvest.

TABLE 3

Effect of ripening temperature and growing area on flavor quality—1955 pack

Analysis of variance

Variation	Mean square	Least significant differences (probability level)	
		.05	.01
Ripening temperature	1346.26***	2.1	2.6
Growing area	84.26***	2.1	2.6
Temperature x area	24.63		
Error	10.03		

***Significant at the 0.001 probability level.

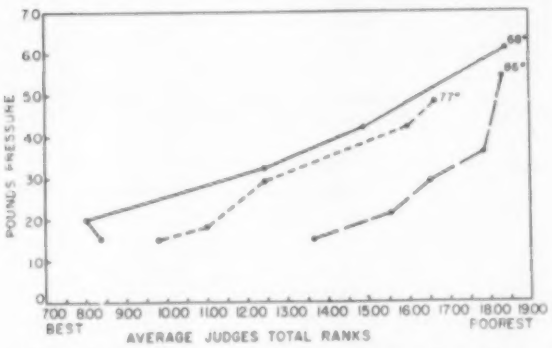

Figure 1. Flavor ranks of Bartlett pears ripened at different temperatures (° F.) and canned at various ripeness levels. Lake County pears, 1954. (Lower ranks indicate highest natural pear flavor intensity).

canned product resulted from pears which were ripened at 68° F. (20° C.) rather than those ripened at 77° F. (25° C.) and 86° F. (30° C.).

Effect of ripeness level. The chemical analyses of fresh and canned Bartlett pears ripened at 68° F. (20° C.) to different pressure tests is shown in Table 5. Within the pressure range studied, acidity of the pears increased and the pH values decreased slightly as the pears ripened. This was true in both the fresh and canned fruit. The Brix/acid ratio of the canned product decreased as the pressure test of the fresh pear decreased. This occurred in the samples from all three different localities. It was due to the increased acidity accompanying the final stages of ripening which more than offset the increase in soluble solids content.

There was a definite trend of increase in VRS, methyl alcohol, and carbonyl compounds with ripening, as shown in Table 6 and Figure 4. Results shown in Figures 1 and 3 and Tables 2 and 4 also indicate the

TABLE 4

Effect of ripeness level and growing area on flavor quality—1955 pack ripened at 68° F. (20° C.)

Analysis of variance

Variation	Mean square	Least significant differences (probability level)	
		.05	.01
Ripeness level	122.06***	0.9	1.2
Growing area	5.66		
Level x area	1.91		
Error	1.97		

***Significant at the 0.001 probability level.

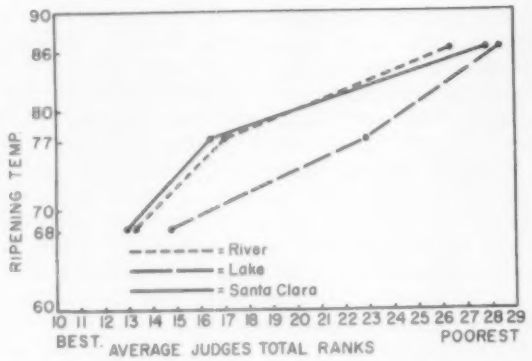

Figure 2. Flavor ranks of canned Bartlett pears from three growing areas ripened at different temperatures (° F.) to 2 lbs. pressure. 1955.

effect of ripeness level at canning on flavor of the canned product. At the same ripening temperature, pears canned at higher pressure test were rated lower in flavor intensity as compared with those canned at lower pressure test. When the pears were canned at 6 pounds pressure, the flavor of the canned product was low, disregarding the difference in temperature during ripening. As the most desirable firmness of Bartlett pears at canning has been shown to be at 2 to 3

TABLE 5

Chemical analyses of fresh and canned Bartlett pears ripened at 68° F. (20° C.) to different ripeness levels—1955 pack

Fresh fruit

Sample	Average pressure test in lbs. (5/16 in. plunger)	Soluble solids as ° Brix at 20° C.	pH	Total acidity as per cent citric
(1) Lake County				
1	3.7	14.85	4.15	.207
2	2.1	14.11	4.10	.195
3	1.5	15.24	3.85	.245
4	1.2	14.52	3.75	.248
(2) Sacramento River District				
5	3.2	12.42	3.95	.288
6	2.4	12.32	3.90	.292
7	1.5	12.54	3.92	.301
8	1.1	12.40	3.90	.328
(3) Santa Clara County				
9	2.9	11.22	4.27	.142
10	2.0	11.61	4.20	.153
11	1.5	11.69	4.13	.160
12	1.1	11.90	4.00	.179

Canned product

Sample	° Brix at 20° C.	pH	Total acidity as per cent citric	Brix/acid ratio
(1) Lake County				
1	17.83±.58	4.30	.111±.009	160.6±5.6
2	17.94±.39	4.24	.113±.003	159.1±5.6
3	18.30±.51	4.09	.138±.003	133.1±2.2
4	18.34±.42	3.94	.140±.011	130.7±1.0
(2) Sacramento River District				
5	16.85±.36	4.06	.185±.004	91.6±2.5
6	16.79±.36	4.08	.181±.003	93.0±1.3
7	16.97±.40	4.03	.183±.019	89.2±1.6
8	17.23±.40	3.91	.188±.024	85.4±1.7
(3) Santa Clara County				
9	16.47±.27	4.51	.084±.005	195.4±4.7
10	16.55±.19	4.45	.087±.004	190.8±7.0
11	16.47±.06	4.32	.091±.005	181.8±9.2
12	16.44±.17	4.28	.092±.003	77.7±5.0

pounds *(11)*, the importance of ripening temperature to natural flavor intensity of the canned product is obvious. Because of this, the change from ambient process to controlled ripening under desirable temperature and humidity is justified, from the standpoint of quality of the canned product.

Effect of growing area. The chemical composition of Bartlett pears from three growing areas, harvested at average pressure tests of 16 to 17 pounds, stored at 32° F. (0° C.), and then ripened at 68° F. (20° C.) to different pressure tests is shown in Table 5. The soluble solids content of the fresh pears from Lake County was approximately 2° Brix higher than those from the Sacramento River District and Santa Clara County at comparable ripeness levels. Pears from the Sacramento River District were higher in total acidity than those from Lake and Santa Clara Counties. The same trends held in the canned products. The differences in chemical composition as illustrated in Tables 1 and 5 can probably be attributed to variations in climatic and soil properties of different growing areas.

The samples from the Sacramento River District and Santa Clara County were higher in total VRS than those from Lake County. Methyl alcohol and carbonyl compounds in the canned product showed the same trend as total VRS content. The differences in VRS content among the samples from different growing areas might be explained by the difference in climatic conditions.

The effect of growing area on flavor of canned pears is illustrated in Tables 3 and 4 and Figures 2 and 3. Although the results do indicate some flavor differences attributable to growing areas, the factors of ripening temperature and ripeness level were more highly significant. It is apparent from the tables that the effects of ripeness level and ripening temperature on flavor of the canned product were the same for all three districts.

DISCUSSION

Pears harvested at an average firmness ranging from 15 to 20 pounds are known to develop best flavor quality after ripening, and mechanical injury is less likely to occur during transportation and handling *(7, 11, 12, 20)*. A canned product of uniform quality is more likely to be obtained if the harvested pears are refrigerated at 41° F. (5° C.) or below for several days. At about 30.2° F. (−1° C.) they may be held without noticeable deterioration for 45 days *(3)*. The pears then may be ripened at 68° F. (20° C.) and 85 to 90 per cent relative humidity to an average pressure test of about 2 pounds. At this stage, the fruit is at optimum ripeness and is best suited for canning *(14)*. The described investigation substantiates the necessity of controlling ripening temperature to improve the quality of the canned product.

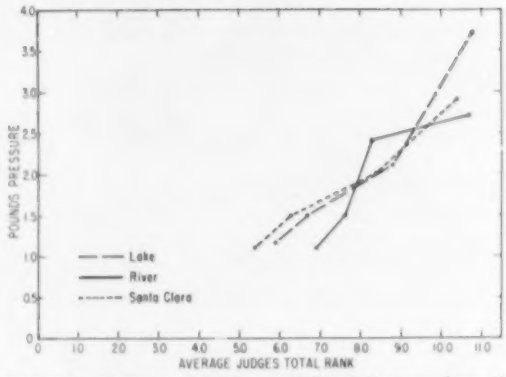

Figure 3. Flavor ranks of canned Bartlett pears from three growing areas ripened to different pressures at 68° F. 1955.

The work of Gerhardt and Ezell *(8)* has indicated an increase of sucrose and fructose and a decrease in total acid during maturation of five commercial varieties of pears on the tree. Dame *et al. (5)* reported the changes in sugars, total acidity, and organic acids in Bartlett pears during ripening at 68° F. (20° C.). A rapid decrease of malic acid and a slower decrease of citric acid during the early stages of post-harvest controlled ripening was found. However, both acids appeared to increase during the late ripening stages. In the present investigation, the total acidity of pears from all three localities was also found to be progressively higher in the later stages of ripening, even somewhat exceeding the acidity at harvest.

The difference in total acidity and Brix/acid ratio among pears from different growing areas is rather pronounced. While the results reported here are not sufficient to draw a general conclusion regarding effect of growing area on chemical composition, it clearly demonstrates the variation of pear composition with variations in climatic conditions. Additional studies on this subject are in progress.

Volatile reducing substances in pears increased greatly during ripening. This was found to be true for pears from all three localities used in this investigation. At comparable firmnesses, the VRS content of pears grown at different localities were not the same (Table 6 and Figure 4); pears grown in the Lake County orchard were appreciably lower at all firmnesses than those from the other localities. It seems likely that there is a yet unknown climatic explanation for this difference in composition. Pears ripened

TABLE 6

Volatile reducing substances in canned Bartlett pears ripened at 68° F. (20° C.) to different pressure tests—1955 pack—(fruit and syrup combined)

Sample	Average pressure test in lbs. (5/16 in. plunger)	Volatile reducing substances, microequiv. $KMnO_4$ per 100 g.	Methyl alcohol p.p.m.	Carbonyl compounds as acetaldehyde, p.p.m.
(1) Lake County				
1	3.7	114	16	2.6
2	2.1	337	27	3.4
3	1.5	520	76	7.9
4	1.2	548	96	7.1
(2) Sacramento River District				
5	3.2	394	36	2.7
6	2.4	510	58	2.7
7	1.5	683	134	7.0
8	1.1	810	366	19.2
(3) Santa Clara County				
9	2.9	493	18	4.6
10	2.0	519	26	5.1
11	1.5	691	29	6.8
12	1.1	721	64	7.4

at 86° F. (30° C.) were lower in VRS content than those ripened at 77° F. (25° C.) and 68° F. (20° C.). The reasons for differences in VRS content are not known, but they could be caused by a modification of the metabolic cycle with temperature, or they could result from a greater volatility of volatile reducing substances at higher temperatures from the fresh fruit during the ripening period.

In a previous publication *(15)*, it was reported that during ripening Bartlett pears increased in total VRS, methyl alcohol, and other compounds with the decrease in pressure test. The rate of production of such volatile compounds was especially rapid when the pressure reading dropped below 2 pounds. However, the presence of large amounts of total VRS, methyl alcohol, and diacetyl in the canned product might be indicative of off-flavor and overripeness at the time of canning. As the results indicated here show that ripening temperatures higher than 68° F. (20° C.) caused a lower VRS, and also lower flavor intensity in the product, it seems likely that relative amounts and chemical composition of flavor constituents in pears may vary with temperature. The highest flavored pears for eating fresh may not necessarily result in the highest flavored canned product. More work is needed to investigate this subject and other constituents which may influence quality of the canned product.

Emmett *(6)* studied the effect of ripening temperature on pectin changes in pears. It was shown that differences in rate of ripening at different temperatures were accompanied by similar differences in the rates at which the pectin changes took place, rapid ripening being associated with rapid decomposition of the pectic substances. Conversion of protopectin to water-soluble pectin is the chief factor concerned in the softening of the fruit after harvest. The work of Dame *et al. (5)* substantiates this work. In the present investigation, fresh pears ripened at 86° F. (30° C.) were observed to be mealy and poor in both texture and flavor with a certain percentage of the fruit failing to ripen properly, while those ripened at 68° F. (20° C.) were juicy. However, the quality of fresh pears changes constantly during ripening. It is difficult to evaluate flavor quality of fresh pears in a manner adaptable to statistical evaluation because of the difficulty in conducting replicate experiments with fresh pears of the same quality. In the canned product, this difficulty is overcome by using a balanced incomplete design method.

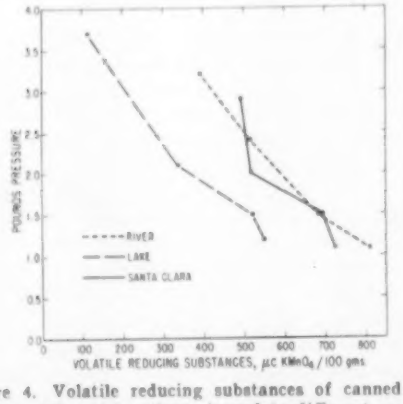

Figure 4. Volatile reducing substances of canned Bartlett pears from different locations ripened to different pressures at 68° F. 1955.

Data obtained in this investigation indicates that Bartlett pears ripened at 68° F. (20° C.) had the highest natural flavor intensity. These results are in agreement with those reported by Ezell and Diehl *(7)* on canned pears, and Hall *(11)*, Pentzer *et al. (18)*, and Tindale and Trout *(20)* on fresh pears. Growing area is of much less importance to flavor than ripening temperature or canning firmness, even though chemical composition is considerably influenced.

Flavor of pears canned at pressure tests of 1.5 and 1.1 pounds were not significantly differentiated by the panel (Table 4). Lower pressure tests of fresh pears at canning seems to be associated with more natural flavor in the canned product. However, pears canned at a pressure test lower than 2 pounds did not result in more natural flavor, because of the undesirable softness *(11)* and possibly undesirable volatile components. Furthermore, pears at this low pressure range are more difficult to handle and are more liable to be rejected on the sorting table during canning.

SUMMARY

Bartlett pears from three of the major pear producing districts in California were harvested at 16 to 17 pounds pressure test, refrigerated at 32° F. (0° C.) for 6 days or longer, ripened at 68° F. (20° C.), 77° F. (25° C.) and 86° F. (30° C.) under 85 per cent relative humidity, and then canned in 25° Brix sucrose syrup at various firmnesses ranging from 3.7 to 1.1 pounds. Results indicate that Bartlett pears from different growing areas differ greatly in content of acid, soluble solids, and volatile reducing substances. Within the pressure range studied, the acidity of the pears increased during the latter stages of ripening, exceeding the acidity at harvest. The volatile reducing substances content in the canned product increased as the firmness of the ripened fruit decreased. Ripening at 86° F. (30° C.) resulted in a slightly higher soluble solids content, a lower VRS content, than ripening at the two lower temperatures, but no significant effect on acidity of the product. Pears ripened at 68° F. (20° C.) to a firmness of about 2 pounds gave a canned product with more natural pear flavor than when ripening to the same firmness was conducted at a higher temperature. Firmness at canning time appeared to be as important as ripening temperature in influencing flavor of the canned product. Growing area had less influence on flavor than the other factors studied. The importance of ripening temperature and firmness at canning to flavor of the canned product is evident.

Acknowledgment

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Storage Changes in Tomato Juice*

B. S. Luh, S. J. Leonard, and G. L. Marsh

Department of Food Technology, University of California, Davis, California

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Quality of tomato juice is largely dependent on the variety and maturity of the fresh tomatoes, methods of processing, and conditions under which the juice is stored. Factors influencing quality of canned tomato juice have been investigated by various workers, who have studied color *(3, 10, 17)*, consistency *(6, 9, 13)*, and vitamin C content *(2, 11)*. The yield and quality of juice from tomatoes graded according to the United States Department of Agriculture Standards have been thoroughly investigated *(4, 7)*. Studies on the effect of storage temperature on vitamin C retention *(1, 5, 15, 19)* have indicated that high storage temperature is undesirable. Comparison of the ascorbic acid content of tomato juices and concentrates stored at different temperatures *(8, 11, 16)* has shown clearly the greater stability of the less concentrated product.

Previous investigations on the stability of tomato juice have dealt mostly with the effect of temperature and duration of storage on vitamin C retention. Exact information on storage life at different temperatures is not clear. There is some discrepancy among reports on the rate at which stored tomato juice deteriorates *(1, 5, 8, 11, 15, 16)*. Vitamin C content of the juice varies with the variety and maturity of the fruit, climatic conditions under which the fruits were grown, and the processing methods. Therefore, storage conditions of canned tomato juice may not be reliably determined by vitamin C content alone. In this work, tomato juice samples were canned under controlled conditions and stored at various temperatures. The samples were analyzed at various time intervals for chemical constituents. It was shown that changes in

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vitamin C, serum color, and amino nitrogen in stored tomato juice may be used to measure objectively the quality of the product. The velocity constant and energy of activation of changes in ascorbic acid and amino nitrogen in tomato juice are presented.

EXPERIMENTAL

Tomato juice canning. Five hundred pounds of Pearson tomatoes at canning maturity were washed, stemmed, sorted, and crushed at room temperature in the Spike hammer-mill type disintegrator. The product was immediately extracted in an American Utensil fruit juice extractor through an 0.033-inch screen, and then deaerated in the James Dole centrifugal deaerator under 29 inches of vacuum. Cannery sodium chloride, 0.65% by weight, was added. The juice was added into No. 2½ plain tin cans with a headspace of ⅜ in., sealed under 15 in. of vacuum, cooked for 30 minutes in a rotary cooker at 100° C. (212° F.), and water cooled. The canned samples were stored at 20° C. (68° F.), 35° C. (95° F.), 45° C. (113° F.), and 55° C. (131° F.) for various lengths of time. At each testing period, samples were drawn for analyses of various chemical constituents, and for flavor evaluation.

Chemical analyses. Analysis of the fresh tomato sample gave the following results: ascorbic acid, 15.2 mg./100 g.; soluble solids, 6.2% at 20° C.; total solids, 7.4%, total acidity, 0.43% as citric acid; and Agtron F color reading, 43.

Ascorbic acid. The 2,6-dichlorophenolindophenol titration method was used to determine ascorbic acid as described previously *(12)*.

Serum color. The samples were filtered through a Whatman No. 2 filter paper, and the color of the filtrate was measured in a Klett-Summerson photoelectric colorimeter, using a blue filter and a distilled water blank. If the serum was turbid and the filtration rate was slow, treatment with pectinol to hydrolyze the pectin was necessary. This was done by first centrifuging the sample to remove the insoluble solids. Then 50 ml. of the centrifugate was treated with 0.1 g. of Pectinol R-10 in a stoppered flask at 40° C. for two hours. The mixture was treated with 0.2 g. of Supercel and filtered through a Whatman No. 2 filter paper. The color of the filtrate was measured as described above. A reagent blank was made from 50 ml. of distilled water, instead of the sample.

Amino nitrogen. Amino nitrogen content of the tomato juice sample was determined by the ninhydrin colorimetric method of Troll and Cannan *(21)* modified as follows: 5 ml. of the tomato juice serum was diluted to 500 ml. with distilled water. Two ml. of the diluted sample was pipetted into a 50-ml. volumetric flask with a glass stopper. To this were added 5 ml. of KCN-pyridine reagent (2 ml. of an 0.01M solution of KCN diluted to 100 ml. with ammonia-free pyridine, which was prepared by shaking 100 ml. of pyridine with 1 g. of permutit for 20 minutes) and 5 ml. of 80% phenol solution (80 g. of phenol dissolved in 20 ml. of absolute alcohol and shaken with 1 g. of permutit for 20 minutes). The flask was stoppered and heated in a boiling water bath for 3 minutes. After partial cooling, 1 ml. of 5% ninhydrin solution in absolute alcohol (w/v) was pipetted into the mixture. The mixture was heated in a boiling water bath for exactly 5 minutes, counting from the time the water started to boil. The solution was cooled in a cold water bath and made up to volume with 60% alcohol. The optical density of the resulting deep blue solution was measured in a Klett-Summerson photoelectric colorimeter, using a green filter. A reagent blank was run, using distilled water instead of the sample. A standard curve was constructed, using glutamic acid concentrations up to 150 μg. per ml. of sample.

Total titratable acidity. Duplicate 10-g. samples of tomato juice were weighed into 250-ml. beakers, and 200 ml. of distilled water was added. The resulting mixture was titrated with 0.1N sodium hydroxide until the pH value reached 8.0 in the Beckman pH meter. The results were expressed as per cent citric acid.

Carotene and lycopene. Carotene and lycopene were determined by a modification of the spectrophotometric method described by McCollum *(14)*. Twenty grams of the sample was diluted to 100 ml. with distilled water and centrifuged. The residue was resuspended in 100 ml. of water and recentrifuged. The residue was extracted several times with a total of about 100 ml. of acetone. The carotenoids were then transferred to 75 ml. of petroleum ether and washed with 50 ml. of water. The petroleum ether extract was treated with anhydrous Na_2SO_4 and then made up to 200 ml. with petroleum ether. A 10-ml. aliquot of the extract was used for chromatography on a column (⅞ in. in diameter and 4 in. tall) made with equal weight of MgO and Supercel. The beta-carotene was eluted from the column with a 30% acetone and 70% petroleum ether mixture (v/v). The xanthophylls were scraped off the top of the column mechanically. Then the lycopene was eluted from the column with a mixture containing 5% ethyl alcohol and 95% petroleum ether. The pigments were measured in a Beckman Model DU spectrophotometer at 472 mμ for lycopene and 450 mμ for beta-carotene.

RESULTS

Rate constant of ascorbic acid deterioration in tomato juice. Tomato juice canned by the process described above was stored at 20° C. (68° F.), 35° C. (95° F.), 45° C. (113° F.), and 55° C. (131° F.) for various intervals of time. The samples were analyzed for ascorbic acid by the 2,6-dichlorophenolindophenol titration method. Initial ascorbic acid content of the canned juice was 14.8 mg./100 ml. Results indicate that ascorbic acid in canned tomato juice was greatly affected by storage temperature. Higher storage temperatures cause a more rapid loss of ascorbic acid. When the per cent retention of ascorbic acid was plotted against time of storage on a semi-log paper, a consistent slope was obtained for each storage temperature. The mechanism of ascorbic acid deterioration may be considered as a first-order reaction.

Table 1 gives the velocity constant and half-life of ascorbic acid in tomato juice stored at various temperatures. For instance, the velocity constant at 55° C. (131° F.) is 0.0239 reciprocal days, i.e., ascorbic acid was decomposing at a rate of 2.39% per day. When the velocity constants at two different temperatures are known, the energy of activation of the reaction can be calculated according to the Arrhenius equation:

$$\log \frac{k_2}{k_1} = \frac{Ea}{2.303R} \cdot \frac{T_2 - T_1}{(T_1 T_2)}$$

Where:

k_2 = velocity constant at temperature T_2
k_1 = velocity constant at temperature T_1
E_a = energy of activation
R = gas constant
T_1 and T_2 = storage temperatures in degrees Kelvin

TABLE 1

Velocity constants of ascorbic acid and amino nitrogen deterioration in tomato juice stored at different temperatures

Temperature of storage		Velocity constant in reciprocal days		Half-life of ascorbic acid
° C.	° F.	Ascorbic acid	Amino nitrogen	
				(days)
20	68	1.27×10^{-3}	0.192×10^{-3}	541[1]
35	95	3.69×10^{-3}	0.705×10^{-3}	188
45	113	7.08×10^{-3}	1.57×10^{-3}	98
55	131	23.90×10^{-3}	6.60×10^{-3}	29

[1] Calculated from energy of activation and velocity constant.

From this equation, using the k values at 35° C. (95° F.) and 45° C. (113° F.), the energy of activation was found to be 12.7 kilocalories per mole.

Darkening of serum color. When the tomato juice samples were stored at high temperatures for a certain period of time, the serum color of the juice remained unchanged for a while, and then started to turn brown rapidly. The degree of darkening may be measured objectively in a Klett-Summerson photoelectric colorimeter, and used as a criterion of storage change. The results on serum color of tomato juice stored at different temperatures are shown in Figure 1. The sample stored at 20° C. (68° F.) for a year did not show any significant darkening. The samples stored at 35° C. (95° F.), 45° C. (113° F.), and 55° C. (131° F.) began to darken after 140, 80, and 15 days, respectively. These values were read from the intersection of the serum color line with the base line for serum color at 20° C. (68° F.) as shown in Figure 1. The serum color of the juice right after canning had a Klett reading of 100 (blue filter, optical density 0.201, and light transmission, 63%. The rate of darkening was more rapid for those samples stored at higher temperatures as indicated by different slopes of the lines. The time in days needed to start the darkening process might be used as an approximate estimation of storage stability at a given temperature.

Decrease in amino nitrogen. Amino nitrogen content of canned tomato juice may decrease on storage. The rate of decrease was especially rapid when the storage temperature was high. Figure 2 indicates the trends of changes in amino nitrogen at different temperatures. The velocity constants of amino nitrogen deterioration at various temperatures are shown in Table 2. Since the rate of decrease in amino nitrogen content at 20° C. is extremely slow, accurate determination of the velocity constant at 20° C. was rather difficult. The energy of activation of this reaction, which can be calculated from the velocity constants at 35° C. (95° F.) and 45° C. (113° F.) was found to have a value of 15.5 kilocalories per mole. From this value the velocity constant at 20° C. was calculated to be 0.192 x 10^{-3} reciprocal days by the Arrhenius equation.

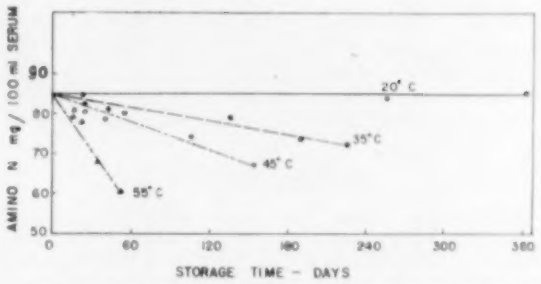

Figure 2. Change in amino nitrogen content of stored tomato juice.

The amino nitrogen content of the juices stored at different temperatures was approximately the same provided they were measured when the serums were just beginning to darken. Thus, both storage temperature and time are important factors influencing the amino nitrogen content. Shorter time and lower temperature favor better retention of amino nitrogen of the juice.

Increase in total acidity. An undesirable metallic flavor and even hydrogen swelling occurred in canned tomato juice stored at high temperatures for a period of time. Hydrogen swelling occurred earlier at higher storage temperatures. This was found to be accompanied by an increase in total titratable acidity. Results in Figure 3 indicate a more rapid increase in total acidity at higher temperatures. No significant increase in acidity was found in a sample stored at 20° C. for a year. Increase in acidity during storage at high temperatures might be related to flavor deterioration and hydrogen swelling. The rate of increase in acidity with time is different at different temperatures. It was a linear function of time at 55° C. (131° F.), but curved at other temperatures. The nature of the acidic compounds formed is not known. They might be formed by degradation or oxidation of reducing sugars present in the juice or by other mechanisms.

Lycopene and carotene retention. The carotenoid pigments in canned tomato juice were found to be

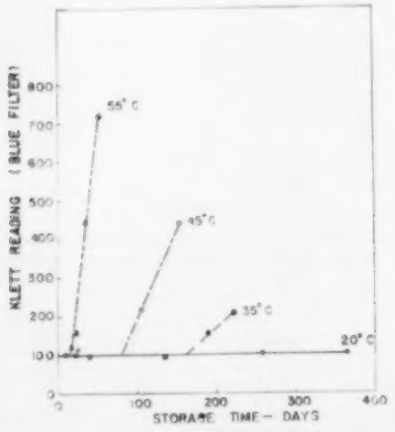

Figure 1. Serum color of stored tomato juice.

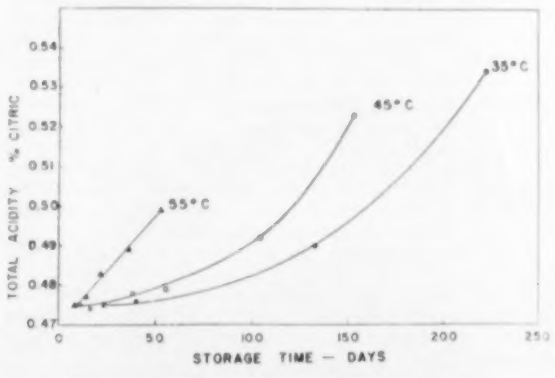

Figure 3. Total acidity of stored tomato juice.

TABLE 2

Decrease of amino nitrogen in canned tomato juice stored at different temperatures

Lot No.	Time needed to start darkening of the serum color, days			Amino nitrogen content, mg./100 g.				Average % loss in amino nitrogen[1]
				At beginning of storage	When serum began to darken			
	35° C.	45° C.	55° C.		35° C.	45° C.	55° C.	
1	174	85.0	19.6	82.5	73.8	72.5	72.6	11.6
2	175	80.0	16.0	79.0	69.0	67.0	70.2	13.0
3	174	75.0	16.0	70.5	63.5	64.5	64.5	9.2
4	180	86.0	19.0	84.0	72.5	73.5	72.6	13.2
5	190	80.0	15.0	82.5	76.0	75.5	78.0	7.3
Average								10.9%

[1] The figures represent the average % loss in amino nitrogen at 35°, 45°, and 55° C. when the serum color was just beginning to darken.

more stable than ascorbic acid and amino compounds during storage at high temperatures. For instance, a tomato juice sample stored at 20° C. (68° F.) for 52 days had a carotene content of 0.63 mg./100 g.; the same sample stored at 55° C. (131° F.) for 52 days had a carotene content of 0.61 mg./100 g. Thus, no appreciable decrease in carotene was found during storage at 55° C. (131° F.) for 52 days, even though the serum had darkened greatly under this condition. The lycopene content of the sample stored at 20° C. (68° F.) was 9.0 mg./100 g.; the same sample stored at 55° C. (131° F.) for 52 days had a lycopene content of 8.0 mg./100 g. These data indicate the stability of carotenoid pigments during storage at high temperatures. The change in color of tomato juice during storage was probably caused by water-soluble, colored compounds formed by the browning reaction, rather than by thermal degradation of the carotenoid pigments. The results reported here agree well with those reported by Guerrant, Vavich, and Dutcher *(5)*.

DISCUSSION

Ascorbic acid has been used as an indicator of storage changes in tomato juice *(1, 5, 15, 19)* because it is unstable towards heat and oxygen. However, difference in conditions under which the juice was made, especially the amount of headspace, oxygen, and copper in the canned product may cause a difference in rate of deterioration of ascorbic acid during storage. In addition, variety and maturity of the fresh tomatoes and climatic conditions under which the tomatoes were grown may also influence the ascorbic acid content of the canned product. Thus, determination of ascorbic acid alone may not tell the storage history of a tomato juice sample, unless the initial ascorbic acid content of the juice is known. However, the rate constant presented in Table 1 might be used to predict the ascorbic acid content under a given storage condition provided the initial ascorbic acid content of the juice is known.

Darkening of the serum color was found to be a simple and reliable indication of storage deterioration. A serum that is darker than usual is evidence of poor storage conditions and poor quality. The time in days needed at a given temperature to start the darkening reaction might be used to estimate the storage stability of the juice at that temperature. Once darkening of the serum starts, deterioration proceeds rapidly, depending on the storage temperature. Although the time needed to start darkening showed the same trend as the half-life of ascorbic acid, the reaction was not brought about solely by the deterioration of ascorbic acid. The decrease in amino nitrogen and possibly other chemical changes may also be involved. For instance, the carbonyl groups of reducing sugars in tomato juice may react with the amino groups of proteins, peptides, and free amino acids to form water-soluble, brown pigments through the Maillard reaction. The decrease in amino nitrogen during storage substantiates this hypothesis.

Decrease in amino nitrogen during storage may be considered as undesirable because of (1) the loss of nutritive value of proteins and amino acids, which become biologically unavailable; (2) the formation of brown pigments, which mask the normal red color of tomato juice; and (3) the formation of compounds of undesirable flavor. It is highly desirable to store tomato juice at lower temperatures, preferably below 20° C. (68° F.), in order to slow down the darkening reaction and to minimize quality deterioration.

Comparison of the velocity constants of decrease in ascorbic acid and amino nitrogen with time indicated different pathways for their deteriorative changes. They differ in energy of activation. Ascorbic acid appeared to be more unstable than amino nitrogen. Possibly both oxidation and thermal destruction are involved in ascorbic acid destruction, whereas amino nitrogen changes mainly through condensation reaction. The mechanism of such changes needs further investigation.

Amino acids in tomato puree have been reported by Safina *(18)* to be glutamine, aspartic, valine, alanine, serine, arginine, glycine, asparagine, leucine, and threonine. Preliminary investigations in our laboratory by paper chromatographic methods have confirmed his results. It remains to be investigated which amino acids are more active in the darkening reaction, and the effect of brown pigment formation on flavor acceptance of the canned product.

It is important to point out that flavor quality of the stored tomato juice was found to be noticeably poor when the decrease in ascorbic acid content was one-half and the loss in amino nitrogen content was 10% of the original value. When the serum color was darker than usual, application of these chemical tests to tomato juice should be useful to the canning industry for quality control purposes.

Increase in total titratable acidity during storage might be one cause of deterioration in flavor. It is not clear whether the dissolution of tin, and eventually

iron, into the serum was caused by high storage temperature or by the formation of acidic compounds that act as tin accepters. Possibly both processes are involved in the hydrogen swelling of cans stored at high temperatures.

The above results show that tomato juice has a relatively short storage life at high temperatures. To preserve the color, flavor, and nutritive value of tomato juice satisfactorily, it should be stored at a temperature of 20° C. (68° F.) or lower.

SUMMARY

Tomato juice was canned under controlled conditions and stored for various lengths of time at 20° C. (68° F.), 35° C. (95° F.), 45° C. (113° F.), and 55° C. (131° F.). The samples were analyzed for various chemical constituents at different time intervals. Storage stability of tomato juice may be measured objectively by the decrease in ascorbic acid and amino nitrogen, and by the darkening of serum color. The half-life of ascorbic acid in canned tomato juices used in this investigation was 29 days at 55° C. (131° F.), 98 days at 45° C. (113° F.), 188 days at 35° C. (95° F.), and 541 days at 20° C. (68° F.). Darkening of the serum color was found to be a simple and reliable indication of storage deterioration. A tomato juice sample with a serum color darker than usual is evidence of poor storage conditions. The time in days required for the serum color to darken at a given temperature might be used to measure the storage stability at that temperature. Amino nitrogen content decreased rapidly when the storage temperature was high. Tomato juice has a relatively short storage life at high storage temperatures. To preserve the flavor, ascorbic acid content, and other chemical constituents, the juice should be stored at 20° C. (68° F.) or lower. The energies of activation and the velocity constants of ascorbic acid and amino nitrogen deterioration in stored tomato juice are presented. The importance of storage temperature to nutritive value, color, and flavor of canned tomato juice is discussed.

Acknowledgment

The authors wish to thank Prof. E. M. Mrak for his guidance and interest in this work. The technical assistance of Mrs. Violet T. Lew and the support of the California Tomato Growers' Association in this investigation are gratefully acknowledged.

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rancidity in mayonnaise for the six-month evaluation period though the controls were perceptibly rancid and faded as early as the third month. The peroxide values confirm the organoleptic and visual tests: in six months the peroxide value of the enzyme treated mayonnaise rose only 0.6 millimole per kilogram as opposed to an increase 24 times as great in the control.

The effect on color is of great importance because of the unfortunate tendency of the housewife to associate mayonnaise color with egg content. Federal standards prevent the use of artificial color so that some mayonnaise manufacturers seek to purchase (often at a premium) dark yolks. The stabilization of the color would tend partially to alleviate the need for deepest color yolks. Uniformity of color and stability of initial color is desired by all manufacturers, and is attainable through the use of the glucose oxidase-catalase.

The amount of gluconic acid formed is negligible (only 200 mg. for the average pint of mayonnaise) and, being bland, has no effect on flavor.

Present Federal Standards do not yet permit the use of Fermcozyme in mayonnaise.

Canned soft drinks. In canned soft drinks oxygen is the usual troublemaker in unsatisfactory shelf life. It is a cause of can perforations, iron pick-up, and fading of product color and flavor.

Tests simulating present canning and warehousing conditions have demonstrated that addition of the Fermcozyme to canned carbonated and non-carbonated beverages will overcome these difficulties caused by oxygen.[b] The enzyme removes the dissolved oxygen as well as that present in the headspace by catalyzing the action between oxygen and glucose to form gluconic acid.

Test packs of grape soda and cola were prepared under conditions simulating good commercial practices in the laboratory of a prominent can manufacturer. Part of the pack was also filled into regular soft drink bottles.

Samples were then stored at room temperature for 10 months.

Addition of enzyme inhibits fading of canned grape

[b] Hammer, Frank and Scott, Don. Increase stability of color and flavor. *Food Processing*. April, 1956.

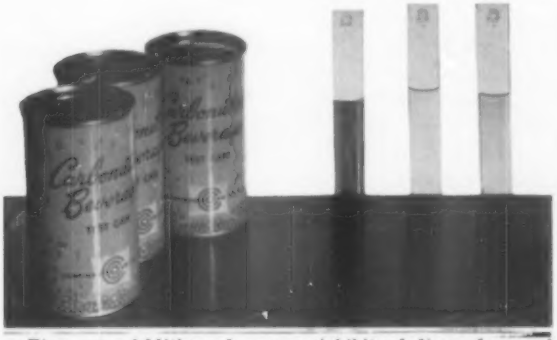
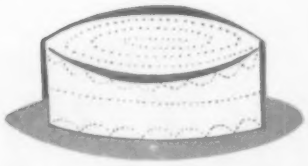


Figure 1. Addition of enzyme inhibits fading of canned grape soda as shown in this black and white photo. The test tube at the left contains soda with 25 units per can and has a purple color. The other two tubes contain soda with no enzyme. The contents of center tube faded to a light green color and the right tube to pale blue, although storage conditions were identical.



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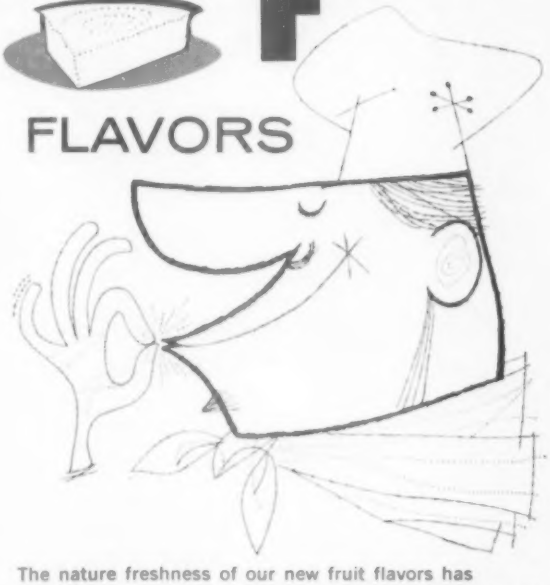

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soda as shown in Figure 1. The test tube at the left contains soda wtih 25 units per can and has a purple color. The other two tubes contain soda with no enzyme. The contents of center tube faded to a light green color and the right tube to pale blue, although storage conditions were identical. There was no noticeable color change in the bottled grape soda. Color of the cola packed in cans did not deteriorate.

Corrosivity tests showed that the Fermcozyme had no effect on any reduceable substance in the soft drinks other than oxygen.

To evaluate flavor, the test packs of canned cola and grape soda were first decarbonated, then tasted and compared against decarbonated bottled samples. There were no clear-cut differences until the 10-month storage period was reached. At this point the grape soda with 0 and 5 units of enzyme was very flat in flavor, while the samples containing 12 and 25 units were indistinguishable from the flavor of bottled samples. In the cola packs no flavor differences in any sample were noted even at 10 months.

Table 2 shows the effect of Fermcozyme level on per cent of unsalable cans due to perforations. Such cans have minute holes or perforations caused by contents reacting with the can wall and result in loss of all or part of the beverage and its carbonation.

TABLE 2

Effect of Fermcozyme on canned grape soda at room temperature; 12 oz. flat-top cans

Units glucose oxidase/can	Per cent perforations			
	4 mo.	5 mo.	6 mo.	10 mo.
0	0	1	18	28
5	0	0	9	15
15	0	0	0	3
25	0	0	0	0

Note that 25 units of enzyme/12 oz. can prevented container perforations during the 10-month test period. Figure 2 shows pickup of iron by these beverages from the can. Note that cans containing the enzyme-treated beverage at 10-month storage had the same iron level as non-treated beverage at 3 months.

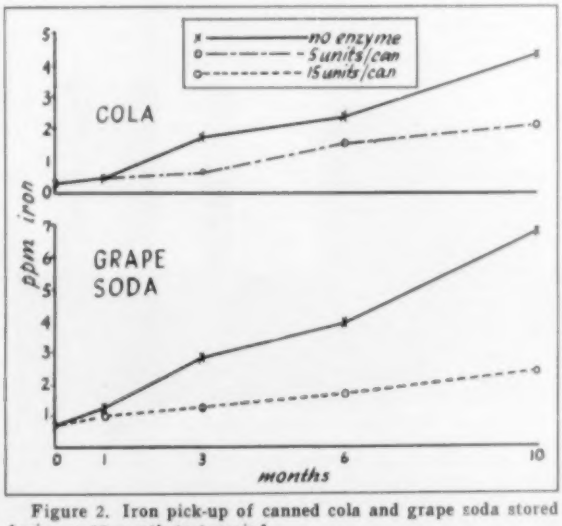

Figure 2. Iron pick-up of canned cola and grape soda stored during a 10-month test period.

Considering all the factors reported in this study (can perforations, iron pick-up, product color and flavor), 15 units of enzyme in a 12 oz. can appears to be sufficient to hold beverage quality and lengthen shelf life of canned grape soda.

As noted in these tests, cola is not susceptible to flavor and color changes when packed in cans as grape soda. Tests still in progress indicate other fruit flavors are similar to grape. Many food scientists have no doubt experienced an instance where a bottle of orange pop left a strong varnish-like aftertaste. This is due to the formation of terpenes, a reaction that takes place by the action of oxygen on exposure of the bottled drink to sunlight. Although in Chicago we do not have the problem to a great extent, this is a very serious problem in warm sunny climates. The incorporation of as little as 2 units of Fermcozyme or 1/400th of a ml. per ounce of orange pop has given complete protection against terpene development even when the flint bottles were shipped South and exposed to sunlight for a week. Under identical conditions the controls were virtually undrinkable after the first day.

In some instances it is possible to include the Fermcozyme in the juice-based flavor concentrate and stabilize it so that the orange concentrate itself is protected against darkening and oxidation, and sufficient activity is carried over to the finished drink to give it full protection too.

In certain applications, where the problem is localized to one surface it is not necessary to mix Fermcozyme in throughout the product to be protected. Thus canned dog food discolors to a grey-brown during retorting at the surface that comes in contact with headspace oxygen. In this instance we have been successful in experimentally preventing this discoloration by surface coating the dog food after filling prior to closing so that the headspace oxygen is removed prior to retorting. This would no doubt be true of other canned meat products as well.

USE AS A SURFACE COATING FOR PACKAGES

Of great interest is the development of packaging materials that have been surface coated with Fermcozyme and dried. In this form the enzyme is inactive. Upon packaging a moist food with this film the enzyme layer picks up moisture from the food and is reactivated. Where necessary, glucose and buffering materials may be incorporated in this surface layer. This deposition may be in solution form or, where the film is dusted with starch, the starch coated with enzyme prior to its use. By using wrappers of this type the entry of oxygen into the loaves of processed cheeses has been prevented.[5] These cheeses tend to form a brown ring that moves in towards the center of the loaf as the oxygen diffuses in. Figure 3 shows loaves of cheese spread that have been stored for 10 weeks at 100° F. and 90% relative humidity. The high humidity tends to make the wrapper more permeable

[5] Unpublished work done with Dr. H. Steinke, Lakeshire Marty Division, Borden Company, and Dr. Don Davis, Milprint, Inc.

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to oxygen so that the brown ring formation is usually noted within 4 to 5 weeks under these conditions. The untreated wrapper is designated "X" while A, B, and C designate high, medium and low enzyme levels, respectively. Note that even the lowest enzyme level tried completely prevents formation of the brown ring. At least one major cheese producer has gone to the expensive foil packaging to control this problem.

One of the major problems facing the supermarkets today is the displaying of prepackaged luncheon meats. Upon exposure to fluorescent lights the meats lose their normal color and turn a disagreeable greyish color. It has been established that this change is due to the action of oxygen under the stimulating effect of the fluorescent lights in the display case. Efforts at using printed cardboard or a metal lid on one side are not of much use because the housewife usually turns over several packages before selecting the one she wants—and leaves the package transparent side up when she leaves. Naturally, the same housewife won't take the package that discolored as a result of her own negligent handling. We have experimented with enzyme coated polycel and indications to date are that the use of a surface coating based on Fermcozyme gives complete protection against this discoloration. This could be a major contribution to packaging if final results bear out the preliminary results.

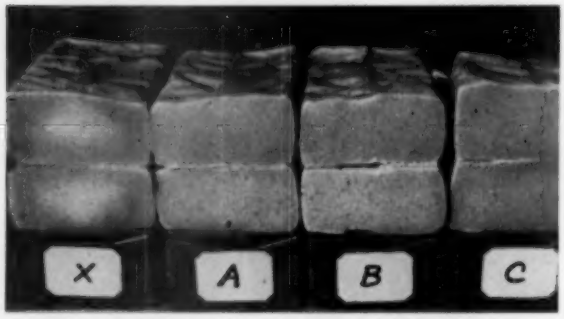

Figure 3. Cheese spread wrapped in Fermcozyme-coated packaging materials, untreated wrapper, X, at left.

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DEOXYGENATION OF DRY FOODS

The latest development in the glucose oxidase picture has been the development of *OxyBan*. *OxyBan* is a dry-appearing, free-flowing granular product that has the ability to take up oxygen. In essence it consists of glucose oxidase-catalase, glucose to act as a substrate, buffers to neutralize the gluconic acid formed that might otherwise limit the reaction, all on an inert edible carrier. *OxyBan* has made possible the application of a rapid means of deoxygenation to dry foods.

Several years ago one of the major soup-mix manufacturers put up dried chicken noodle soup for the Armed Forces. Specifications called for gassing to reduce the oxygen level below 2%, and, at the time of packing, the oxygen level was that low. But when time came for delivery it was found that the oxygen level had increased to over the 2% level because of the release of dissolved oxygen from the noodles. The soup mix had to be repackaged. Now no amount of gassing or vacuum treatment that is economical will get out dissolved oxygen completely. That is why the specification, requiring the removal of 90% of the oxygen, is considered a tough one. That is why when one talks of a "high vacuum pack" for something like coffee we talk of only 27 in. of vacuum. With *OxyBan*, it is possible routinely to reduce the oxygen in a container to below limits detectable on an Orsat apparatus, for *OxyBan* will continue to work—after the package is sealed—until all the oxygen is exhausted, provided of course sufficient glucose and buffer are present.

Tests run by placing *OxyBan* in an empty can and sealing have shown that the oxygen content as determined by Orsat analysis can be reduced below 0.25% within 2 hours. Of course, this requires a rather high enzyme level and work that we have done with commercial products has been with an enzyme level designed to deoxygenate an empty can in 18–24 hours. This time will be affected, of course, by the nature of the product packed in the can. If it is a product like potato chips or dried chow mein noodles, the time should be about the same as for an empty can, but with a product like dried whole milk the physical impeding of the diffusion of oxygen within the mass will slow the overall removal.

While *OxyBan* is edible, it is not what would be termed "tasty"—it's more like eating flavorless dry cereal. Hence, we do not recommend its direct incorporation into foods for human consumption. In some

instances, such as animal feeds, this direct incorporation has a pronounced protective effect on the carotene, when the alfalfa meal is packaged in a fairly impermeable outer package.

Of wider applicability is the use of *OxyBan* as a packet. This packet is analogous to a packet of desiccant but instead of taking up moisture, it takes up oxygen. Coffee protected with *OxyBan* appears to do as well as high vacuum packed coffee. Smoked yeast, which loses its flavoring value very rapidly, may be kept at full strength for months with *OxyBan* packets. That *OxyBan* can take up oxygen completely has been demonstrated. It now remains only to apply it to those products known to deteriorate through the action of oxygen.

The acceptability of Fermcozyme and *OxyBan* as food ingredients is well established because of the extensive use of a sister product, Ovazyme, glucose oxidase-catalase, for the stabilization of egg solids. In this instance, the enzyme system is used in a somewhat similar form to remove glucose, with oxygen being present in excess. Literally tens of millions of pounds of whole egg, yolk and egg white solids have been consumed by the civilian and military in this country. Toxicity tests conducted at the time this work was started demonstrated no deleterious effect on repeated feedings of the enzyme system or the egg products made with it.

The progress made in the packaging field in the development of inexpensive films that act as good oxygen barriers complements the work that has been done on enzymatic oxygen scavengers to give products a shelf life that would have been unheard of a few years ago.

Together they make a perfect team; the enzyme system can remove the oxygen within the package, and a good package can keep out any further oxygen. Together, they can give perfect protection.

ABSTRACTS

—Compiled by H. A. Campbell

ANALYTICAL METHODS

Separation of aliphatic volitile acids by paper chromatography.

OSTEUX, R., GUILLAUME, J. AND LATURAZE, J. (Inst. Pasteur, Lille, France). *J. Chromatography 1*, 70–84 (1958).

The volatile aliphatic acids from C_1 to C_6 are chromatographed on paper after recovery by steam distn. under controlled conditions, elimination of cations and concn. by passage over a sulfonated resin. The acids are transformed into their morpholine salts and chromatographed on Whatman No. 3 paper in the solvent-system BuOH/cyclohexane/propylene glycol/NH_3 22° Be/morpholine/H_2O (30: 30: 10: 0.7: 0.07; 3.5) for the sepn. of the straight chain acids, and in the system benzyl alc. satd. with 1.5 *N* ammonia for the "iso" series. Revelation is carried out by spraying with an alc. soln. of *o*-cresolsulfonphathalein in sodium veronal buffer. The method has given excellent results, notably in the detn. of the fermentation type or pattern of anaerobic bacteria.

Separation and identification of aliphatic mercaptans by chromatography of the 2,4-dinitrophenylsulfides.

CARSON, J. F. AND WONG, F. F. (Western Utilization Research and Devel. Div., Albany, Calif.). *J. Org. Chem. 22*, 1725–6 (1957).

Separation of 2,4-dinitrophelsulfides of some of the aliphatic mercaptans by adsorption chromatography on silicic acid contg. fluorescent zinc sulfide is described. The rates of movement of alkyl dinitrophenylsulfides were found to increase in the order: $CH_3 < C_2H_5 < CH_2 = CH - CH_2 < nC_3H_7 < nC_4H_9 < nC_5H_{11} < tC_4H_9 < nC_6H_{13}$. Mixtures of any of these derivs. could be resolved into sep. distinct bands which could be isolated as pure compds.

Rapid sedimentation of proteins through starch.

ANDERSON, N. G. Biol. Div., Oak Ridge Natl. Lab., Tenn.). *Nature 181*, 45–6 (1958).

Comparisons revealed that during centrifugation protein moves through starch at about twice the rate observed in free soln. This phenomenon is caused by the formation of small zones of higher protein concn. which would tend to decant off the starch grains and flow between them until collision with another starch grain occurred. These findings may be applied to equilibrium centrifugation and to sedimentation of large molecules such as deoxyribonucleic acid.

BIOLOGICAL SCIENCE

Biochemistry

Terminal steps of chlorophyll a biosynthesis in higher plants.

WOLFF, J. B. AND PRICE, L. (Div. of Radiation and Organisms, Smithsonian Inst., Washington, D. C.). *Arch. Biochem. Biophys. 72*, 293–301 (1957).

In a number of common higher plants, the formation of chlorophyll a has been found to require a dark, enzymic reaction following a photochemical step. Solubility and chromatographic data indicate that protochlorophyllide is photochemi-

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cally reduced to chlorophyllide a, which is then esterified with phytol, in the presence of chlorophyllase, to form chlorophyll a.

Studies on the amide and C-terminal residues in proteins 3. The esterification of proteins.

Chibnall, A. C., Mangan, J. L. and Rees, M. W. (Dept. of Biochem., Univ. of Cambridge, Engl.). *Biochem. J. 68*, 114–18 (1958).

Methods of esterifying proteins have been investigated in detail. Full esterification can be achieved with methanolic 0.1 N-HCl in 24 hrs., in confirmation of the results of earlier workers. The hydrolytic loss of amide nitrogen is, however, much greater than has heitherto been reported. Data are presented to show the relative rates of esterification of aspartyl, asparaginyl and glutamyl residues in insulin and β-lactoglobulin.

Ultracentrifugal determination of molecular weight of myosin by the Archibald procedure.

Mommaerts, W. F. (Dept. of Med., Univ. of California, Los Angeles). *Science 126*, 1294 (1957).

Measurements were made on 4 different crystd. myosin prepns. using a Spinco ultracentrifuge equipped with an optical system for the observation of Rayleigh interference fringes. The mol. wt. was consistently found to be 382,000 with individual variations within ± 20,000. A figure of 380,000–400,000 corresponds to the presumable wt. of the myosin moiety of the 504,000 g. of actomyosin which reacts stoichiometrically with 1 mole of ATP. Hence, the myosin molecule has one active center for this interaction.

The synthesis of serum albumin and tissue proteins in slices of rat liver and liver tumor.

Campbell, P. N. and Stone, N. E. (Courtauld Inst. of Biochem., Middlesex Hosp. Med. School, London, Engl.). *Biochem. J. 66*, 19–31 (1957).

The prepn. of rat-serum albumin with ammonium sulfate fractionation and zone electrophoresis on columns of 'cellulose' is described. The homogeneity of the albumin was investigated by boundary electrophoresis and immuno-electrophoresis. A net synthesis of serum albumin occurred during the incubation of slices of liver and liver tumor from rats bearing liver tumors induced by feeding on 4-dimethylaminoazobenzene. From a comparison of the results obtained with liver slices and liver-tumor slices it is concluded that the hepatoma cells in liver tumors retain the ability of the liver cells to synthesize serum albumin. Determination of the absolute amt. of serum albumin synthesized by slices indicate that the tumor slices synthesize only 30–50% as much albumin as do a similar wt. of liver slices.

The utilization of phenylalanine and tyrosine for protein synthesis by human cells in tissue culture.

Eagle, H., Piez, K. A. and Fleischman, R. (Natl. Insts. of Health, Bethesda, Md.). *J. Biol. Chem. 228*, 847–61 (1957).

The metabolic utilization of uniformly C^{14}-labeled phenylalanine and tyrosine by 3 human cell strains in tissue culture has been examd. Both amino acids were rapidly incorporated into cell protein in the absence of net protein synthesis, from medin lacking one or more essential amino acids other than glutamine. With all 3 cell lines, 0.7 to 1% of the phenylalanine or tyrosine residues of the protein was replaced by the labeled amino acid of the medium per hr. This rate was sustained for 3 days, and was approx. halved by the omission of glutamine from the medium.

Enzymic synthesis of L-ascorbic acid in different animal species.

Grollman, A. P. and Lehninger, A. L. (Dept. of Physiol. Chem., Johns Hopkins Univ. School of Med., Baltimore, Md.). *Arch. Biochem. Biophys. 69*, 457–67 (1957).

The 3 enzymes or enzyme systems involved in the biosynthesis of L-ascorbic acid from D-glucuronic acid in animal tissues have been assayed in exts. of various tissues of a no. of different animal species. In mammals requiring no dietary ascorbic acid, the liver contains all 3 enzymes and thus can convert D-glucuronate to L-ascorbate. The kidney contains the first 2 enzymes but is apparently unable to carry out the last step. In mammals requiring dietary ascorbic acid, including man, 2 monkey species and the guinea pig, the liver is unable to convert L-gulonate to ascorbate, although enzymes are present.

A study of the chemical, physical, and kinetic properties of intestinal lipase.

Dinella, R. R. (Vanderbilt Univ., Nashville, Tenn.). *Dissertation Abstr. 17*, 1654 (1957).

Some of the chem., phys., and kinetic properties of intestinal lipase were investigated using cell-free homogenates of hog duodenal mucosa. Of the natural lipids tested only glycerides were hydrolyzed by intestinal lipase. The optimal pH was 9–10. Calcium ions and bile salts stimulate activity. Inhibitors were studied.

Microbiology

Mechanisms of action of antibiotics. 1. Additive action of chloramphenicol and tetracyclines on the growth of Escherichia coli.

Ciak, J. and Hahn, F. E. (Dept. of Rickettsial Diseases, Walter Reed Army Inst. of Research, Washington, D. C.). *J. Bacteriol. 75*, 125–9 (1958).

The actions of chloramphenicol, chlortetracycline, and oxytetracycline in subbacteriostatic concns. on the growth rate of *Escherichia coli* B were found to be additive. Equimolar concns. of chlortetracycline and oxytetracycline elicited nearly identical growth-inhibitory effects while, molecule for molecule, chloramphenicol was approx. 5 times less active. The mechanisms of action of the 2 tetracyclines are considered to be identical. The additive effect of chloramphenicol and tetracyclines is interpreted as concurrent blocking of different metabolic pathways which contribute to protein synthesis.

Necrogenic potency of yeasts grown on various media.

Gitler, C., Sunde, M. L. and Baumann, C. A. (Dept. of Biochem., Coll. of Agr., Univ. of Wisconsin, Madison). *J. Nutrition 63*, 399–408 (1957).

Death due to hepatic necrosis occurred rapidly and in 100% incidence in vitamin E-depleted rats fed *Torulopsis utilis* grown on spent sulfite liquor as the sole source of protein. Comparable diets contg. brewers' by-products yeast did not produce necrosis. *Torulopsis utilis* and *Saccharomyces cerevisiae* grown on a synthetic medium were both found to be necrogenic, while these same yeasts grown on com. beer wort were both found to be non-necrogenic. Liver necrosis was prevented by the addn. of methylene blue or diphenyl-*p*-phenylenediamine to the diet.

A continuous freeze drier for laboratory studies.

Rhian, M., Maister, H. G. and Hutton, R. S. (Northern Utilization Research and Devel. Div., Peoria, Ill.). *Appl. Microbiol. 5*, 323–31 (1957).

The construction and operation of a continuous freeze drier suitable for prepg. up to 60 g. of dry material per hr. are described. Material that is frozen in the form of pellets is fed into the drier and the dry pellets are removed without interrupting the drying process. The process may be controlled and adjusted as required by variations in the diameter of the pellets, rate of, sublimation, density of the load in the drier, the solids content of the pellets and the desired moisture content of the final products. The drier is small enough so that many trials may be completed and large enough so that sufficient material may be prepd. for adequate measurement of critical properties of the dried prepns.

Nutrition

Chronic undernutrition and starvation in the rat.

Nutrition Revs. 15, 148–50 (1957).

Starvation produced a greater relative loss of body fat in young rats than did undernutrition. During starvation, the livers of young male rats lost more wt., in terms of per cent than did the rest of the body; liver glycogen disappeared in adults during starvation but was maintained during undernutrition; for all groups, the loss of liver nitrogen in starvation was greater than in undernutrition; there was only a slight loss of skeletal tissue in both starved and undernourished adult rats, and in the young rats during starvation, whereas the formation of skeletal tissue was reduced in the young undernourished rats.

Amino acids feeding in kwashiorkor.

Nutrition Revs. 15, 132–3 (1958).

Evidence presented suggests that the amino acid pattern, rather than the nitrogen content alone, may be the limiting nutrient of the diets of children in which kwashiorkor develops. The "initiation of cure" response can occur in children receiving a mixt. of 18 amino acids as their source of nitrogen. Quantitative and qual. amino acid requirements remain to be detd. for both the prevention and cure of kwashiorkor.

The absorption of vitamin B_{12} in normal and gastrectomized rats and the effect of some gastric extracts.

Clayton, C. G., Latner, A. L. and Schofield, B. (Dept. of Radiotherapy, Royal Victoria Infirmary, Engl.). *Brit. J. Nutrition 11*, 339–45 (1957).

Radioactive vitamin B_{12} was given orally to normal and gastrectomized rats and absorption detd., from estn. of fecal radioactivity. For a const. dose of 16 mμ g. vitamin B_{12} normal rats showed a mean absorption of 42.9 ± 1.0%. In gastrectomized animals it was reduced to 5.5 ± 1.1%. Rat gastric juice given simultaneously with the vitamin produced no change in the absorption in normal rats, but raised that of the gastrectomized rats to 15.9 ± 1.2%. The results suggest the possible production of an intrinsic factor by the rat stomach.

Experimental hypervitaminosis A in man.

Nutrition Revs. 15, 134–5 (1957).

Effects of administration to a 40 yr. old human of 1,000,000 I.U. of vitamin A in aq. emulsion daily for 13 days and an addnl. 2,000,000 I.U. on the 14th day produced the following toxic symptoms: dermatitis, epistaxis, gastrointestinal disturbances, thinning of the hair, visual disturbances, muscle weakness, pain and tenderness over the long bones, dizziness, and increased fragility of the fingernails. Symptoms appeared within 3 days. Repetition of the above expt. 5 months later for a 25 day period produced less severe symptoms. The author suggests a measure of adaptation may have been responsible for symptom reduction in the later test.

The effects of adequate and excessive calcium when fed with adequate phosphorus in growing rations for beef calves.

Dowe, T. W., Matsuoshima, J. and Arthaud, V. H. (Univ. of Nebraska, Lincoln). *J. Animal Sci. 16*, 811–20 (1957).

Two 140-day feeding trials were conducted to compare the effects of adequate calcium with excess calcium on the performance of beef calves fed adequate phosphorus in a growing ration. Excess calcium appeared to depress gains by interfering with the digestibility and/or absorption of nutrients and mineral elements other than phosphorus.

The role of fat in the diet of rats. 12. Effect on rats of type and quantity of dietary fat with and without linoleate supplementation.

Funch, J. P., Jorgansen, E. and Dam, H. (Dept. of Biochem. and Nutrition, Polytechnic Inst., Copenhagen. *Brit. J. Nutrition 11*, 426–33 (1957).

The influence of increasing dietary levels of trilaurin or hydrogenated arachis oil on the requirement for essential fatty acids in male rats was investigated. The rats received the exptl. diets for a 26-wk. period from weaning. Hydrogenated arachis oil depressed growth and damaged the testes, kidneys and skin to a higher degree than trilaurin at corres. dietary levels. The deleterious effect of hydrogenated arachis oil could be partly due to the presence of isomers of the unsatd. fatty acids formed during hydrogenation. The noxious effect of the fat was counteracted by supplementation with 20 mg. ethyl linoleate/animal/day and completely prevented by 100 mg./animal/day.

Nutrition studies in the cold. I. Influence of diet and low environmental temperature on growth and on the lipid content in the rat.

Treadwell, C. R., Flick, D. F. and Vahouny, G. V. (Dept. of Biochem., Med. School, George Washington Univ., Washington, D. C.). *J. Nutrition 63*, 611–21 (1957).

Immature and young adult male rats received a series of a 8 cholin- and inositol-free diets varying in protein content from 5 to 40% (with 20% fat) and in fat from 10 to 40% (with 20% protein). At 25° the liver lipids in the immature rats were highest on the 5% diet and dropped regularly and sharply up to 30% protein. At 1° the liver lipids were essentially the same at all protein levels and were in the normal range for the rat. Increasing the fat content of the diet from 10 to 40% produced a regular increase in liver lipids at 25° while at 1° the normal range at all fat levels. The observations on the young adult animals were qualitatively the same as for the immature rats. The dietary requirement for lipotropic factors appeared to be lower at 25° for the adult animals.

Metabolic adaptations in higher animals. I. Dietary effects on liver glucose-6-phosphate.

Freedland, R. A. and Harper, A. E. (Dept. of Biochem., Univ. of Wisconsin, Madison). *J. Biol. Chem. 228*, 743–51 (1957).

Rats fed diets in which protein, fat, galactose, or fructose was substituted for a direct glucose source showed a marked increase in liver glucose-6-phosphatase activity calcd. per g. of liver, per unit of body wt., or per g. of liver protein. Pair feeding decreased the differences between the control and exptl. groups; however, the differences remained highly significant. The feeding of a protein-free diet for 4 days had no significant effect on the liver glucose-6-phosphatase activity of rats that consumed the diet readily.

Biological value of proteins.

Nutrition Revs. 15, 151–3 (1957).

Rate of release of amino acid in digestion has been suspected as having a bearing on the nutritive value of protein. Conditions were established for the *in vitro* digestion of proteins with pepsin, trypsin and erepsin. Studies proved the pattern of amino acids vary as the acids are released in digestion. Concentrations of essential amino acids in pepsin-digests and in completely hydrolyzed proteins were detd. The calcn. of the percentage of each amino acid in the pepsin digests and other indices are described. Comparison of the amino acid indices of several proteins as calcd. in this expt. with their biol. values, as detd. in the rat-feeding procedure, indicated very good agreement. This *in vitro* procedure may prove reliable in predicting the nutritional quality of proteins.

Caloric value and composition of foods.

Nutrition Revs. 14, 306–8 (1956).

Caloric value of most foods have been made on the basis of their chem. compn., by using conversion factors for the content of crude protein, ether ext. and carbohydrate (by difference). Many carbohydrates included in this fraction are of no value to man or other non-ruminants. Available carbohydrates defined as starches, dextrins, di- and monosaccharides give more realistic caloric values. A recently reported adaptation of the

anthrone method provides a simplified method for detn. of available carbohydrate. Considerable differences are found in calorie values calcd. on the basis of carbohydrate by difference and available carbohydrates.

Prevention of coprophagy in the rat.

Barnes, R. H., Fiala, G., McGehee, B. and Brown, A. (Graduate School of Nutrition, Cornell Univ., Ithaca, N. Y.). *J. Nutrition 63*, 489–97 (1957).

A technique has been described for the totnl prevention of coprophagy in the rat. Measurement of the extent to which rats maintained on raised wire screens eat their feces indicated that 50 to 65% of the excreted feces are normally ingested. In the presence of a nutritional deficiency, fecal consumption may be even greater.

FOOD AND FOOD TECHNOLOGY

Baking and Bakery Products

Fractionation and reconstitution procedures for cake flours.

Sollars, W. F. (Western Wheat Quality Lab., Agr. Research Service, Pullman, Wash.). *Cereal Chem. 35*, 85–99 (1958).

Bleached and unbleached cake flours were sepd. into water-sols., gluten, tailings starch, and prime starch by an acetic acid fractionation procedure. Cake flour fractions could be successfully reconstituted by drying of the dough, grinding, and rehydration. The reconstituted flours gave angel-food cakes matching closely the vols. of the cakes from the original flours. Good white layer cakes were obtained from all fractionations with other acids than acetic, but only citric acid fractionation resulted in yellow layer cakes comparable with those from acetic acid fractionations.

The content of amino acids in white flour and bread.

McDermott, E. E. and Pace, J. (Cereals Research Sta., St. Albans, Engl.). *Brit. J. Nutrition 11*, 446–52 (1957).

Contents of phenylalanine, tyrosin and serine in bread hydrolysate were each lower, to the extent of about 5–7%, than in flour hydrolysate. The content of phenylalanine, tyrosin and lysine was 15–16% less in crust hydrolysate than in flour hydrolysate. In discussing the significance of these losses, attention was drawn to the difficulty of establishing unequivocally how far they may be due to the baking process itself and how far to destructive action during hydrolysis, which may vary from flour to crust.

Bread flavor.

Wiseblatt, L. Chem. Natural Food Flavors Symp. U. S. Quartermaster Food and Container Inst. (May 1957); *J. Sci. Food Agr. 9*, i–45 (1958).

The flavor as with all processed foods is a function of the ingredients and of the processing. The following are discussed: roles of ingredients, fermentation, oven reactions, and flavor compds. detected in bread. Gas chromatography has been used to identify very small amts. of volatile substances. (14 references).

Cereals and Grains

The improvement of the protein quality of white rice by lysine supplementation.

Rosenberg, H. R. and Culik, R. (Stine Lab., E. I. du Pont de Nemours and Co., Inc., Newark, Del.). *J. Nutrition 63*, 477–87 (1957).

The nutritive value of the protein of white polished rice can be improved significantly by supplementation with the first limiting amino acid, lysine, in amts. sufficient to bring this amino acid in balance with the second limiting amino acid. Substantial improvements in growth rate and in efficiency of food utilization were obtained with 0.05 to 0.10% of L-lysine HCl · supplementation. This growth response was accompanied by a decrease in the amt. of fat and by an increase in the amt. of protein deposited in the liver.

Fats and Oils

Brown-colored oxypolymers of unsaturated fats.

Venolia, A. W. and Tappel, A. L. (Univ. of California, Davis). *J. Am. Oil Chemists' Soc. 35*, 135–8 (1958).

The oxypolymerization of unsatd. fat in the presence of protein and amino acid was studied to det. whether the active carbonyl-amine browning reaction is a dominant mechanism for the formation of brown-colored polymers. A highly reactive system of menhaden oil-aqueous egg albumin emulsion was studied in greatest detail. Three types of studies: activation energy of formation of brown color, inhibition analysis of the mechanism leading to formation of brown color, and measures of amino acid reaction in the oxidizing unsatd. fats, all gave evidence that active carbonyl-amine browning was not a dominant mechanism.

The present status of acetoglycerides.

Alfin-Slater, R. V. and Coleman, R. D. (Univ. of Southern California, Los Angeles). *J. Am. Oil Chemists' Soc. 35*, 122–7 (1958).

Acetoglycerides, which are defined as being either triglycerides contg. 2 acetyl groups per mol. or diglycerides contg. one acetyl group, can be prepd. readily by several procedures, which are described. Fairly extensive investigations of the physiol. behavior of acetoglycerides have been made. It is concluded, from the work reported to date, that acetoglycerides have no deleterious effect on growth or on reproduction and that the mode of digestion of acetooleins and acetostearins does not differ appreciably from that of the corres. natural fats. A no. of potential uses of acetoglyceride products as components of foods and cosmetic prepns. are mentioned. One possible use of acetostearins appears to be as a protective coating for food products.

Feedstuffs

The influence of certain antibiotics on the palatability of swine rations.

Tomlin, D. C., Wallace, H. D. and Combs, G. E. (Florida Agr. Expt. Sta., Gainesville). *J. Animal Sci. 17*, 42–6 (1958).

Chlortetracycline enhanced the palatability of swine rations and was much preferred, when a choice was given, over oxytetracycline, penicillin V and erythromycin. Oxtetracycline and penicillin V did not clearly improve or depress the acceptability of feeds contg. them. Pigs completely avoided feed contg. erythromycin when other feed was available.

Effect of trichloroethylene-extracted meat scrap on young cattle.

Rehfeld, C. E., Perman, V., Sautter, J. H. and Schultze, M. O. (School of Veterinary Med., Univ. of Minnesota, St. Paul). *J. Agr. Food Chem. 6*, 227–30 (1958).

Calves fed meat scrap produced by azeotropic extn.-dehydration with trichloroethylene developed a moderate to severe thrombocytopenia and relative lymphocytosis and, in one instance, leucopenia. These effects are similar to those induced in young cattle by feeding certain specimens of trichloroethylene-extd. soybean oil meal. Development of toxic properties in cattle feeds processed with trichloroethylene is not restricted to soybeans.

Fish

Canning of fish in stainless steel "EI-457."

Grzhivo, V. S. and Syunyakova, Z. M. *Konserv. i. Ovoshchesushil.* Prom 12, No. 8, 26–9 (1957); *Chem. Abstr. 52*, 3191 f (1958).

Several kinds of fish (some prepd. in tomato sauce, in oil, and as is) were canned in stainless steel "EI-457" and (as a control) in tin. The products were stored for 3–4 years and analyzed yearly for Fe and Cr. During the storage period the Fe concn. did not change. The

Cr concn. in the products stored in stainless steel was 8–9 times greater than that in the control and increased with the time of storage.

Fruits and Vegetables

The effect of in-package desiccation on the storage properties of compressed and uncompressed dehydrated cabbage.

Gooding, E. G. B. and Duckworth, R. B. (Ministry of Agr., Aberdeen, Scotland). *J. Sci. Food Agr. 9*, 14–18 (1958).

The rate and extent of water loss from compressed cabbage was rather less than from uncompressed material, but the rate of deterioration of uncompressed and compressed material appeared to be generally similar. Desiccation reduced the rate of deterioration in flavor, texture and ascorbic acid content, as well as in color.

Gelatin

Purity of gelatin as a protein.

Russell, G. (Chem. Research Lab., Ilford Ltd., Brentwood, Engl.). *Nature 181*, 102–3 (1958).

Chromatography on ion exchange resin columns was used to assess the purity of gelatin samples. This procedure divided the gelatin into 2 fractions which were examd. qualitatively by paper chromatography. This showed that the protein resembled serum albumin and differed from gelatin in that it contained no hydroxyproline and a low concn. of glycine. The variability of tyrosin content of gelatin is discussed in the light of recent expts. by the author and other workers.

Chemically distinct major components of gelatin.

Russell, G. (British Gelatine and Glue Research Assoc.). *Nature 181*, 537–8 (1958).

In an attempt to sep. impurities from gelatin, expts. were carried, in which chromatographic column techniques were applied, using a finely divided form of the resin, 'I.R.C. 50'. At pH 5.5 only a portion of the sample (15%) is eluted, the remainder (85%) being firmly adsorbed on the resin. Paper chromatograms showed the smaller component to have a higher tyrosin content than ordinary gelatin, no detectable hydroxyproline and other characteristics giving it an amino-acid compn. broadly similar to the serum albumins. Comments from the assocn. members indicated that the future course of research on gelatin may be very much modified by this work.

The biuret complex of gelatin and the mechanism of gelation.

Bello, J. and Vinograd, J. R. (California Inst. of Technol., Pasadena). *Nature 181*, 273–4 (1958).

By the use of the biuret reaction, it was shown that the peptide group plays an important part in gelation. Formation of the biuret complex of gelatin caused reversible suppression of gelation. Destruction of the complex by acetic acid resulted in instantaneous gelation at 0°. These results show that the inhibition of gelation is due to the copper-peptide complex and not to side chain complexes. Reasons for inhibition of gelation are discussed in addn. to the nature of the copper binding by the gelatin.

A fundamental investigation of adhesion. II. Method for measuring shrinkage stress in restrained gelatin films.

Weatherwax, R. C., Coleman, B. and Tarkow, H. (Forest Products Lab., Forest Service, U. S. Dept. of Agr., Madison, Wis.). *J. Polymer Sci. 27*, 59–66 (1958).

A technique is described for measuring the shrinkage stress within the plane of a homogeneously restrained gelatin film. At 50% relative humidity, stresses of 4000 to 5000 lbs. per square inch were observed in several films. The reality of these stresses was confirmed by a completely independent method.

Influence of time on the reversible association between large molecules; the collagen system.

Gross, J. (Dept. of Med., Harvard Med. School, Boston, Mass.). *Nature 181*, 556 (1958).

This communication reports an example of time-related changes in soly. of fibrils formed in relatively homogeneous solns. of collagen. On cooling a gel for 10 min. after it had reached max. opacity at 37° C., there was fairly prompt fall in opacity, leveling off at about 40% of the control value. The loss in opacity on cooling after 10 min. incubation was 100% and after 48 hrs. of incubation was less than 10%. Opacity, in this system was a direct function of pptd. protein sedimentable at 80,000 g., for 1 hr. It is suggested that the increasing degree of irreversibility may result from the action of Brownian movement, allowing the assocd. mol. to find their most stable and sterically matched fit, thus permitting the establishment of more intermol. cross-links.

Milk Technology

Growth and reproduction of rats on diets of evaporated milks and a vegetable fat milk product.

Ney, L. F. (Biol. Dept., Stanford Research Inst., Menlo Park, Calif.). *J. Agr. Food Chem. 6*, 223–7 (1958).

Growth and reproduction studies were carried out with rats to compare the nutritive value of an exptl. vegetable fat milk product with a conventionally processed and a high-temp., short-time processed evaporated milk. Judged by the criteria of wt. gain and reproduction performance, the milk product made from nonfat milk solids, vegetable fats, and water, and fortified with vitamins, was somewhat better nutritionally than the other 2 milks. The poorest performance was shown by the rats fed the conventional evaporated milk, which appeared to be inadequate to meet the needs of female rats during repeated gestation and lactation.

Factors affecting the germicidal property of raw milk.

Henningson, R. W. (Cornell Univ., Ithaca, New York). *Dissertation Abstr. 16*, 1559–60 (1956).

Raw cows milk will prevent or inhibit the growth of many micro-organisms. As a result of this study it was concluded that a deficiency of free sulfhydryl groups and/or denatured lactalbumin is a major cause of the germicidal property.

Tea

Studies on the 'fermentation' of Ceylon tea. X. Pectic enzymes in tea leaf.

Ramaswamy, M. S. and Lamb, J. (Tea Research Inst., Talawakelle, Ceylon). *J. Sci. Food Agr. 9*, 46–51 (1958).

Fermenting tea leaf produces MeOH as the result of enzymic demethylation of pectins contained in the leaf. The enzyme concerned (which was isolated and purified) is very similar to pectin-methylesterases found in other plants. The activity of tea pectin-methylesterase is partially inhibited by tea catechins and their oxidation products, but nevertheless a continuous demethylation process gives rise to pectic acid during the course of fermentation.

Miscellaneous

Problems of the experimenter.

Youden, W. J. (Natl. Bureau of Standards). *Ind. and Eng. Chem. 50*, 89A–90A (1958).

It is demonstrated that a sampling pattern, which leads to min. deviation (due to an unmeasured variable) between 2 test procedures, may well lead to increased variation in the individual results of each of the test procedures.

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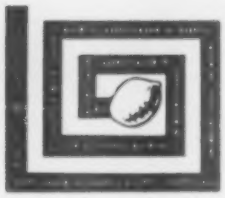

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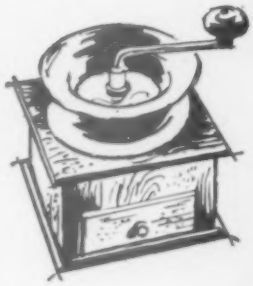

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